P.CIT.	For re	eceiving Office use only
PTOREGE® 07 JUL 200	φ	
e de	International Application	No.
REQUEST	International Filing Date	
The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.	Name of receiving Office	e and "PCT International Application"
	Applicant's or agent's file (if desired)(12 characters mu	
Box No. I TITLE OF INVENTION GYPSUM PRODUCT		
Box No. II APPLICANT		
Name and address: (Family name followed by given name; for designation: The address must include postal MOBIL OIL COMPANY LIMITED	a legal entity, full official code and name of country.)	This person is also inventor.
Mobil House 500-600 Witan Gate		Telephone No.
Central Milton Keynes Buckinghamshire MK9 1ES		Facsimile No.
United Kingdom		Teleprinter No.
State (that is, country) of nationality: GB	State (that is, country)	of residence:
		e United States America only the States indicated in the Supplemental Box
Box No. III FURTHER APPLICANTS AND/OR (FUR		
Name and address: (Family name followed by given name; for designation. The address must include postal	a legal entity, full official code and name of country.)	This person is:
BOELEE, Scotia 105 Forest Road		applicant only
Tunbridge Wells		X applicant and inventor
Kent TN2 5BG		inventor only (if this check-box
United Kingdom	•	is marked, do not fill in below.)
State (that is, country) of nationality:	State (that is, country)	of residence: GB
This person is applicant all designated all designa		e United States America only the States indicated in the Supplemental Box
Further applicants and/or (further) inventors are indicated		America only — the dayperson
Box No. IV AGENT OR COMMON REPRESENTATI		R CORRESPONDENCE
The person identified below is hereby/has been appointed to ac of the applicant(s) before the competent International Authoritie	et on behalf	gent common representative
Name and address: (Family name followed by given name; for designation. The address must include postal	z legal entity, full official code and name of country.)	Telephone No.
Gill Jennings & Every		+44 171 377 1377
Broadgate House		Facsimile No.
7 Eldon Street	•	+44 171 377 1310
London EC2M 7LH		Teleprinter No.
United Kingdom		(051) 22765 GILPAT G
Address for correspondence: Mark this check-box whe	re no agent or common re	presentative is/has been app inted and the

*		i i	i Ti	No.	X
			l (ra-		
W. Fe'			** : *	¥	
		, F. 1			1
	. 7				
		- 4.		-	. 4
			24.	. The	
		, and the second	**		
	-2		* .	,	
	The state of the s	* .			1
	and the second s				}
		=			*
		į.			
					TO.
		***	F		
	¥.	· · · · · · · · · · · · · · · · · · ·	,		
	i Alas Kale ayan baran da ka	a company of the second	and the second of the second o		1
	•		* * * * * * * * * * * * * * * * * * * *		
		1-1		3 / /w	
		•		* *	
		± " • =			
		V			
		C	· • · · · · · · · · · · · · · · · · · ·		
		1	Hope .		
	* •				
				and the second	-
•	**				
			5.4		
The state of the s					*
			200		
4"		•			
	and the second s	Y			
\$4.			e e e e e		ż
			* v		4
				en en	
					*



Box No.V DESIGNATION OF STATES

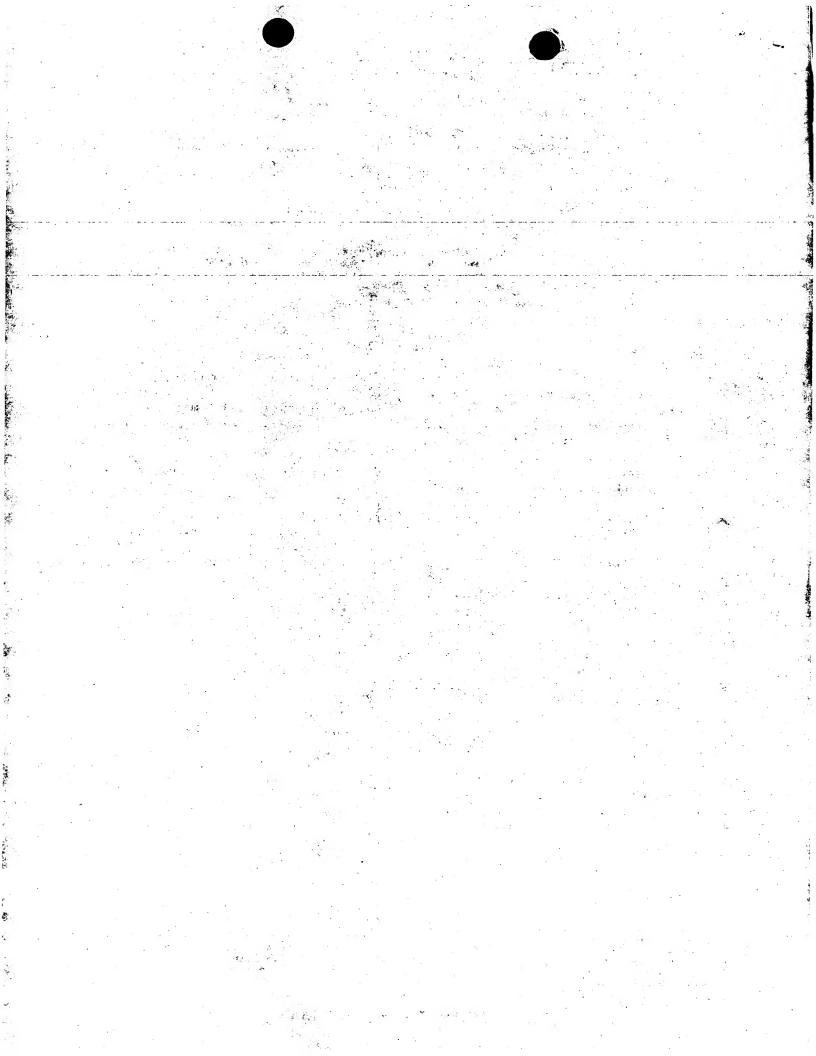
The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes; at least one must be marked):

Regional Patent

- AP ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SZ Swaziland, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- EA Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyztan, KZ Kazakstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- EP European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- OA OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT (if other kind of protection or treatment is desired, specify on dotted line)

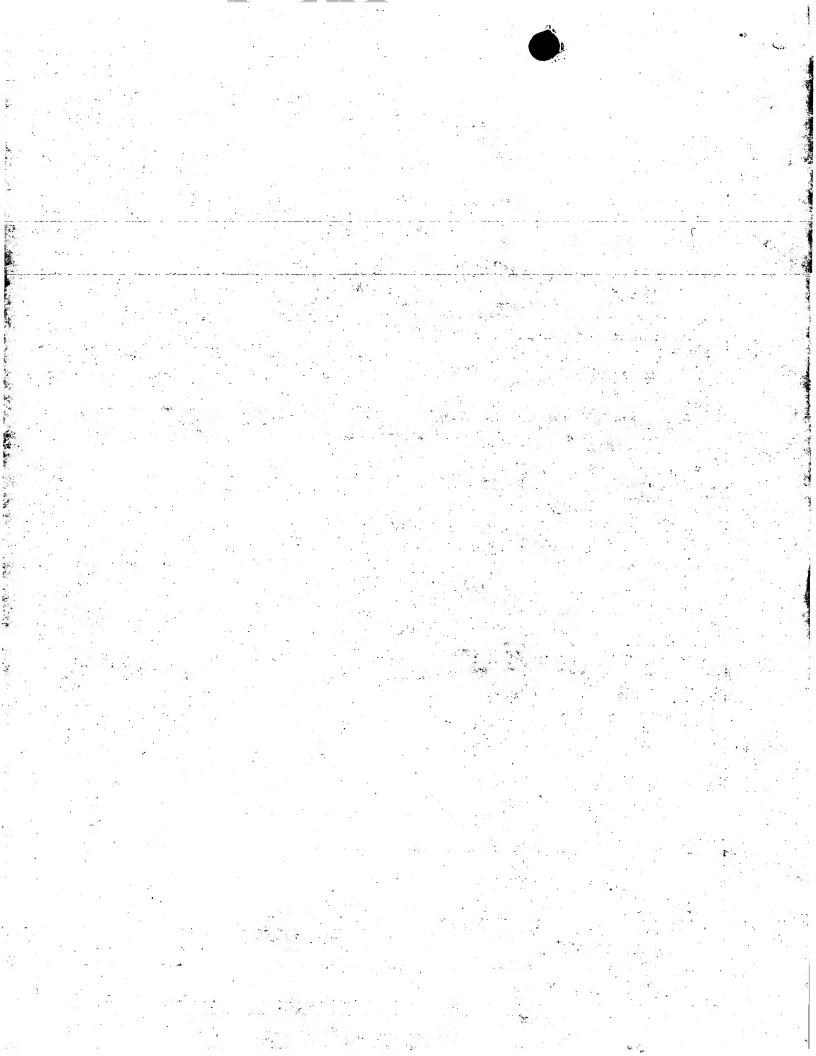
Nation	al Pat	ent (if other kind of protection or treatment is desired, s	pecify on	dotted line):	
X	AL	Albania	X	LS Lesotho	•
X	AM	Armenia	X	LT Lithuania	
X		Austria	\mathbf{X}	LU Luxembourg	
X	ΑU	Australia	X	LV Latvia	
X	AZ	Azerbaijan	X	MD Republic of Moldova	
X	BA	Bosnia & Herzogovina	X	MG Madagascar	
X	BB	Barbados	X	MK The former Yugoslav Republic of Macedonia	
X	BG	Bulgaria			
X	BR	Brazil	X	MN Mongolia	
X	BY	Belarus	X	MW Malawi	
X	CA	Canada	X	MX Mexico	
X	CH	and LI Switzerland and Liechtenstein	X	NO Norway	
[X]		China	X	NZ New Zealand	
X		Cuba	X	PL Poland	
X	C7.	Czech Republic	<u> </u>	PT Portugal	
X		Germany	X	RO Romania	
X		Denmark	X	RU Russian Federation	
X		Estonia	<u> </u>	SD Sudan	
X	ES		X	SE Sweden	
X		Finland	X	SG Singapore	
X		United Kingdom	<u> </u>	SI Slovenia	
X .		Georgia	X	SK Slovakia	
X		Ghana	X	SL Sierre Leone	-
(X)		Gambia	X	TJ Tajikistan	_
		Guinea-Bissau	X	TM Turkmenistan	
	7.00	Croatia	X	TR Turkey	
X		Hungary	X	TT Trinidad and Tobago	
X		Indonesia	X	UA Ukraine	
<u>X</u>	IL.	Israel	X	UG Uganda	
	_		<u>X</u>	US United States of America	
X	IS JP	Iceland Japan	LΔI	OS United States of America	
X		,	X	UZ Uzbekistan	
_		•	X	VN Viet Nam	
X		Kyrgyzstan	X	YU Yugoslavia	
X	KP	Democratic People's Republic of Korea			
_			X	ZW Zimbabwe	
X		Republic of Korea	Check	ck-boxes reserved for designating States (for the purposes	of
\boxtimes		Kazakstan	a nati	tional patent) which have become party to the PCT after ince of this sheet:	
X		Saint Lucia			
X	LK	Sri Lanka	X	.GDGrenada	
X	LR	Liberia	X	.INIndia	. ,

Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation of a designation consists of the filing of a notice specifying that designation and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit.)



	,
3	

Box No. VI PRIORITY C	LAIM	Further priority claims are indicated in the Supplemental Box.					
Filing date	Number	Where earlier application is:					
of earlier application (day/month/year)	of earlier application	national application: country	regional application:* regional Office	international application: receiving Office			
item (1) 08/01/1998	9800368.4	GB					
08 January 1998	,						
item (2)			11.				
item (3)							
The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the Receiving Office) identified above as item(s):							
* Where the earlier application is Convention for the Protection of In	an ARIPO application, it is m	andatory to indicate in the Si	upplemental Box at least o	ne country party to the Paris			
	NAL SEARCHING AU		u (Rute 4.10(0)(ii)). Bee b	appienamus Dox.			
Choice of International Search (if two or more International Search competent to carry out the internat	ching Authorities are sear	quest to use results of earl	ier search; reference to r requested from the Inter-	that search (if an earlier national Searching Authority).			
the Authority chosen; the two-letter	r code may be used): Dat	e (day/month/year)	Number	Country (or regional Office)			
ISA/							
Box No. VIII CHECK LIST							
This international application cor the following number of sheets:	tains This internationa	l application is accompanies ation sheet	d by the item(s) marked l	pelow:			
request :	3 1 = 1	igned power of attorney		·			
description (excluding sequence listing part)	13 3. copy of g	eneral power of attorney; re	eference number, if any:				
claims	4. statement	explaining lack of signature	e	. •			
abstract :	1 5. priority d	ocument(s) identified in Box	x No. VI as item(s):				
drawings :	2 6. translation	n of international application	n into (language):				
sequence listing part of description :	0 = '	ndications concerning depose and/or amino acid sequence					
Total number of sheets :	9. \(\) other (spe	•	Ŧ -	*			
Figure of the drawings which should accompany the abstract:	Lar	guage of filing of the rnational application:	ENGLISH				
Box No. IX SIGNATURE	OF APPLICANT OR AG	ENT					
Next to each signature, indicate the nan	ne of the person signing and the c	apacity in which the person sign	s (if such capacity is not obvio	ous from reading the request).			
For the Applicant Gill Jennings & Eve	ery						
			. •				
JONES, Helen Marjor	rie Meredith		Date: 08 Janu	uary 1999			
	Ec. 7	ceiving Office use only					
Date of actual receipt of the international application:		Serving Office use only a		2. Drawings:			
Corrected date of actual rectimely received papers or determined the purported international	lrawings completing			received:			
Date of timely receipt of the corrections under PCT Arti	e required cle 11(2):			not received:			
International Searching Aut (if two or more are compet)			al of search copy delayed fee is paid	ed			
	For Inter	rnational Bureau use only					
Date of receipt of the record c	ору						

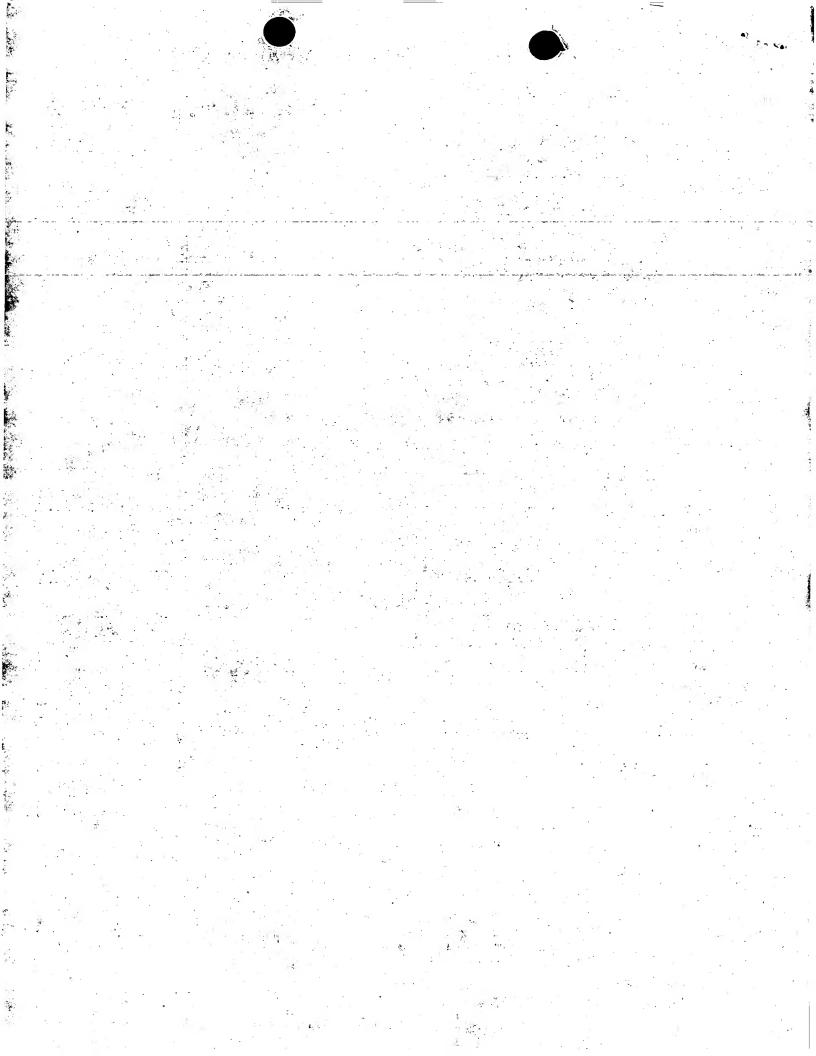


PCT	For receiving Office use only
FEE CALCULATION SHEET Annex to the Request	International Application No.
Applicant's or agent's file reference HMJ03045WO	Date stamp of receiving Office
Applicant MOBIL OIL COMPANY LIMITED	
CALCULATION OF PRESCRIBED FEES 1. TRANSMITTAL FEE 2. SEARCH FEE International search to be carried out by (If two or more International Searching Authorities are competent in relation application, indicate the name of the Authority which is chosen to carry out it is international application contains 23 sheets. First 30 sheets	£ 22.00 P £ 1824.00
deposit account (see below) cheque cash postal money order revenue stamps	other (specify):
DEPOSIT ACCOUNT AUTHORISATION (this mode of payment may The RO/ is hereby authorized to charge the total fees is is hereby authorized to charge any deficiency deposit account.	

Signature

Date (day/month/year)

Deposit Account Number



PATENT COOPERATION TREAT Rec'd 07 JUL 2000 From the RECEIVING OFFICE To: Gill Jennings & Every **Broadgate House** NOTIFICATION OF THE INTERNATIONAL APPLICATION NUMBER AND OF THE 7 Eldon Street INTERNATIONAL FILING DATE London 29 JAN 1999 (PCT Rule 20.5(c)) EC2M 7LH GILL JENNINGS & EVERY Date of mailing 26.01.99 (day/month/year) Applicant's or agents's file reference IMPORTANT NOTIFICATION **HMJ03045WO** International application No. International filing date (day/month/year) Priority date (day/month/year) PCT/GB99/00064 08/01/1999 08/01/1998 Applicant Mobil Oil Company Limited et al Title of the invention **Gypsum Product** The applicant is hereby notified that the international application has been accorded the international application number and the international filing date indicated above. The applicant is further notified that the record copy of the international application: 26.01.99 was transmitted to the International Bureau on has not yet been transmitted to the International Bureau for the reason indicated below and a copy of this notification has been sent to the International Bureau*: because the necessary national security clearance has not yet been obtained. because (reason to be specified): The International Bureau monitors the transmittal of the record copy by the receiving Office and will notify the applicant (with Form PCT/IB/301) of its receipt. Should the record copy not have been received by the expiration of 14 months from the priority date, the International Bureau will notify the applicant (Rule 22.1(c)).

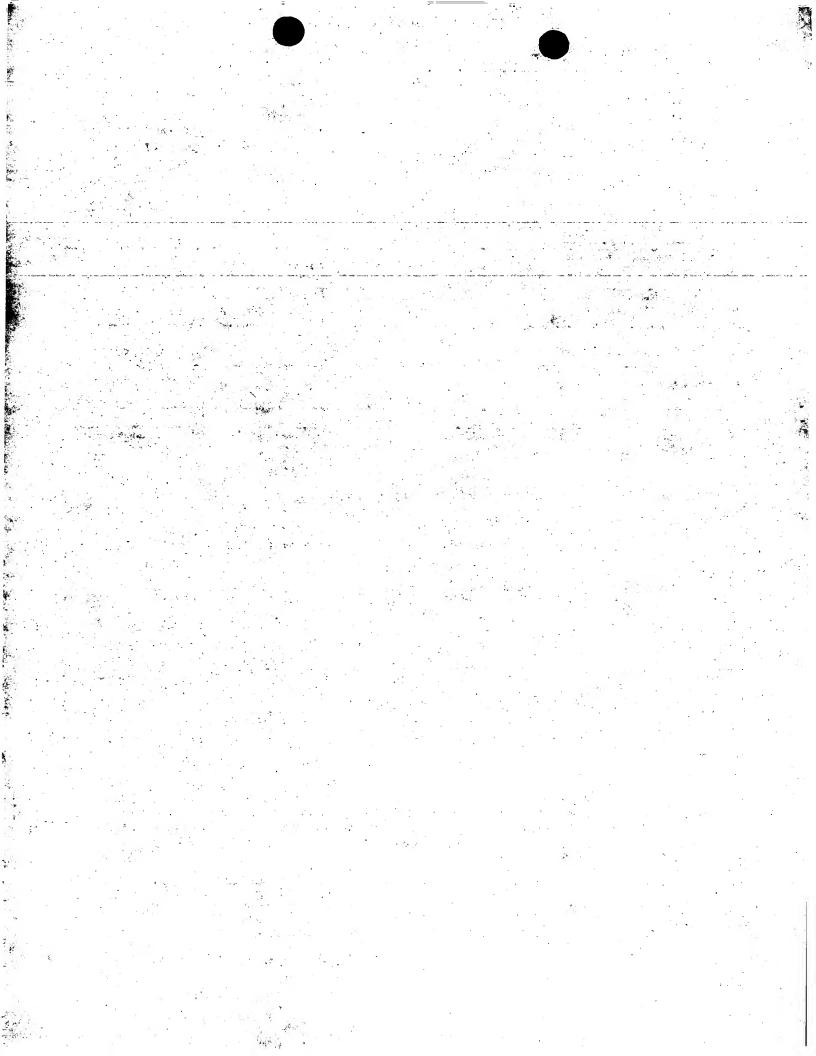
Name and mailing address of the receiving Office
The Patent Office
Cardiff Road, Newport
South Wales NP9 1RH

Facsimile No.

Authorized office
PHIL TREEN

Telephone No. 01633 814381

Form PCT/RO/105 (July 1992)



PTO/PCT Rec'd 0 7 JUL 2000

NOTIFICATION CONCERNING SUBMISSION OR TRANSMITTAL OF PRIORITY DOCUMENT

(PCT Administrative Instructions, Section 411)

From the INTERNATIONAL BUREAU

7 Eldon Street London EC2M 7LH ROYAUME-UNI

GILL JENNINGS & EVER 1 3 APR 1995

GILL JENNINGS & TYTINY

Date of mailing (day/month/year) 31 March 1999 (31.03.99)

Applicant's or agent's file reference HMJ03045WO

International application No. PCT/GB99/00064

International publication date (day/month/year)

Not yet published

IMPORTANT NOTIFICATION

International filing date (day/month/year) 08 January 1999 (08.01.99)

Priority date (day/month/year)

08 January 1998 (08.01.98)

Applicant

MOBIL OIL COMPANY LIMITED et al

- The applicant is hereby notified of the date of receipt (except where the letters "NR" appear in the right-hand column) by the International Bureau of the priority document(s) relating to the earlier application(s) indicated below. Unless otherwise indicated by an asterisk appearing next to a date of receipt, or by the letters "NR", in the right-hand column, the priority document concerned was submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b).
- This updates and replaces any previously issued notification concerning submission or transmittal of priority documents.
- An asterisk(*) appearing next to a date of receipt, in the right-hand column, denotes a priority document submitted or transmitted to the International Bureau but not in compliance with Rule 17.1(a) or (b). In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.
- The letters "NR" appearing in the right-hand column denote a priority document which was not received by the International Bureau or which the applicant did not request the receiving Office to prepare and transmit to the International Bureau, as provided by Rule 17.1(a) or (b), respectively. In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.

Priority date

Priority application No.

Country or regional Office or PCT receiving Office

Date of receipt of priority document

08 Janu 1998 (08.01.98)

9800368.4

GB

29 Marc 1999 (29.03.99)

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland

Authorized officer

Juan Cruz

Telephone No. (41-22) 338.83.38

Facsimile No. (41-22) 740.14.35

				:					
				• 10		·= .			
4					201	1			
<i>⊃</i> ≱⊢				1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	. **	i i			
5	•						• ***		
				, ·					
				·. 28					<u>.</u>
	*		65	• 00	•			*	
		0.2						0,	
43	9	1 1944	i Januar Ayara		-10				
						1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	4	* * *	
·	16						w.y.		
					Adams comment of the state of t	كسيوخ بإدويها ف	<u> </u>	چهند څخه د مونځ	
									- 6
*	May 1	• 6 0				to the second			
9	ا المعقدة والمستوال المسترارية المراجعة المسترارية المعتقدة المسترارية المسترارية المسترارية المسترارية المسترارية		and the same of the same of	ب معدمات بأنبالوساساريد		ing and a second			2
	A second second						*		
						deservices.			
2					r e	*-			9
	A			3-T		·			
*				1	· ·		۵		
4	10 7 mg	e o see						3.1-	
25			. 1		1 T A P		A	B11	
•	3. + 40	Contract of the second				Birth Free		•	
		AND HORSE AND AND AND ADDRESS.					* *	* 1	
		Sec.							
	And the second of the second		- P						·
44. #	4.5			- CK					
- 1				10 T			1. 30		
50.4			R 44						,
1000									
								, 45.	_
			*						
						A de la companya de l			
5 S. C.						Article			
									e e e e e e e e e e e e e e e e e e e
									and the second s
である。 これでは、これでは、これでは、これでは、これでは、これでは、これでは、これでは、									

PATENT COOPERATION TREATY

TO/PCT Rec'd 07 JUL 2000

INFORMATION CONCERNING ELECTED OFFICES NOTIFIED OF THEIR ELECTION

(PCT Rule 61.3)

From the INTERNATIONAL BUREAU

To:

GILL JENNINGS & EVERY Broadgate House 7 Eldon Street London EC2M 7LH ROYAUME-UNI

Date of mailing (day/month/year)

26 January 2000 (26.01.00)

Applicant's or agent's file reference

ı

IMPORTANT INFORMATION

HMJ03045WO
International application No.

PCT/GB99/00064

International filing date (day/month/year) 08 January 1999 (08.01.99)

Priority date (day/month/year)
08 January 1998 (08.01.98)

Applicant

MOBIL OIL COMPANY LIMITED et al

 The applicant is hereby informed that the International Bureau has, according to Article 31(7), notified each of the following Offices of its election:

AP :GH,GM,KE,LS,MW,SD,SZ,UG,ZW

EP:AT,BE,CH,CY,DE,DK,ES,FI,FR,GB,GR,IE,IT,LU,MC,NL,PT,SE

National :AU,BG,BR,CA,CN,CZ,DE,IL,JP,KP,KR,MN,NO,NZ,PL,RO,RU,SE,SK,US

2. The following Offices have waived the requirement for the notification of their election; the notification will be sent to them by the International Bureau only upon their request:

EA:AM,AZ,BY,KG,KZ,MD,RU,TJ,TM

OA:BF,BJ,CF,CG,CI,CM,GA,GN,GW,ML,MR,NE,SN,TD,TG

National :AL,AM,AT,AZ,BA,BB,BY,CH,CU,DK,EE,ES,FI,GB,GD,GE,GH,GM,HR,HU,ID,

IN,IS,KE,KG,KZ,LC,LK,LR,LS,LT,LU,LV,MD,MG,MK,MW,MX,PT,SD,SG,SI,SL,TJ,TM,

TR,TT,UA,UG,UZ,VN,YU,ZW

3. The applicant is reminded that he must enter the "national phase" before the expiration of 30 months from the priority date before each of the Offices listed above. This must be done by paying the national fee(s) and furnishing, if prescribed, a translation of the international application (Article 39(1)(a)), as well as, where applicable, by furnishing a translation of any annexes of the international preliminary examination report (Article 36(3)(b) and Rule 74.1).

Some offices have fixed time limits expiring later than the above-mentioned time limit. For detailed information about the applicable time limits and the acts to be performed upon entry into the national phase before a particular Office, see Volume II of the PCT Applicant's Guide.

The entry into the European regional phase is postponed until 31 months from the priority date for all States designated for the purposes of obtaining a European patent.

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland **Authorized officer:**

Olivia RANAIVOJAONA

Telephone No. (41-22) 338.83.38



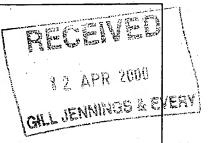
e and the		also the same of t	-
		, E.	
. C.		**************************************	1800
-			
		•	
		2	•
24.			
		:	
		-5.1	
1 1 1 m			
£"			
	가 되는 것은 사람들이 되었다.		
Ľ	agaighealagh an a' mar ag agus 1914 agus an agus ag agus gar an an ag an ag ar ag a		
45			
			₹.
9			
4.0			
	그렇게 하는 사람들이 얼마나 나는 사람들이 되었다. 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그	Ř.	. 4
10			3
		*	1,3
		- 100	
· A			
Ċ			
1.5	이 경기 사람이 사용하는 것 없어요. 그는 사람들이 되었다고 나는 사람들이 가지 하는 것 같아. 그렇게 되었다.	N. marin	3.7
		111	

			129
Ţ.	그는 그는 사람들(아니라) 그 아이들은 아이들이 그는 사람들이 하는 사람들이 가장 사람들이 되었다. 그 사람들이 아니라 가장하다.		7.
	[조하뉴 1.] 그렇게 가는 사람이 가장 된 시간에 되었다. 그 그 그 그 그 그 그 사람들은 사람들이 가장 가고 있다고 있다.		
€ -	하는 바다 하다 하다 하다는 회사들이 하는 그는 사람들은 사용 없다. 하는 데 보다는 그는 사람들에 하는 하는 사람들이 하는 것을 하는 것 같다.		* 4
4	그는 이렇게 하는 그 전에 살았다. 그렇게 되었는데 하는데 하는데 하는데 하는데 하는데 하는데 하는데 하는데 하는데 하		
3	사고하다 보다 하고 있는데 그리고 있다면 하고 있다. 그 그 그 그 그 그 그 그 그 그 그 사람이 되는 사람이 되었다. 그 그 그 사람이 사람이 사람이 사람이 없다.		煮
4 5	그는 보는 소문으로 그렇게 하는 그 그 그 그는 그들은 사람들이 있는 것이 그 사람들이 모든 수가는 그는 것을 모든 것을 다 되었다.		
10.	이 가는 아이들이 아이들 때문에 가는 사람들이 되었다.		
is.	하는 우리는 물문이는 얼마나 하는 사람들은 사람들이 가는 사람들이 가는 사람들이 되었다. 그 사람들이 되었다.		
			14
· · · · · · · · · · · · · · · · · · ·			
· · · · · · · · · · · · · · · · · · ·			
(1) · · · · · · · · · · · · · · · · · · ·			
· · · · · · · · · · · · · · · · · · ·			
· · · · · · · · · · · · · · · · · · ·			
では、 100mmの 100mm 100m			
を受けている。 1965年の日本の日本の日本の日本の日本の日本の日本の日本の日本の日本の日本の日本の日本の			
· · · · · · · · · · · · · · · · · · ·			
(1) 1 (1)			
を開発している。 できない はない かんかん できない かいかい しゅうかん はない かいかい かいしゅう かいかい しゅうしゅう かいかい かいかい かいかい かいかい かいかい かいかい かいかい かい			
を受けている。 「そのではないのでは、「ないでは、「ないでは、「ないでは、「ないでは、「ないでは、「ないでは、「ないでは、「ないでは、「ないでは、「ないでは、「ないでは、「ないでは、「ないでは、「ない いっぱん しょうしゅう しょうしゅう しゅうしゅう しゅう			
を開発している。 できる できない かんしゅう かんしゅう はいかい かいかい かいかい かいかい かいかい かいかい かいかい かいか			
 			
《《···································			
を思われています。 こうこう はい かいかい かいかい かいかい かいかい かいかい かいかい かいかい			
《《《··································			
《《···································			
《《···································			
新聞館 1 分割 1 分			
を受けている。 「それではないのでは、これでは、これでは、これでは、これでは、これでは、これでは、これでは、これ			



INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

GILL JENNINGS & EVERY Broadgate House 7 Eldon Street London EC2M 7LH **GRANDE BRETAGNE**



PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY **EXAMINATION REPORT**

(PCT Rule 71.1)

Date of mailing (day/month/year)

10.04.00

IMPORTANT NOTIFICATION

Applicant's or agent's file reference HMJ03045WO

International application No. PCT/GB99/00064

International filing date (day/month/year)

Priority date (day/month/year) 08/01/1998

08/01/1999

Applicant

MOBIL OIL COMPANY LIMITED et al.

- 1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
- 2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
- 3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/

Fax: +49 89 2399 - 4465

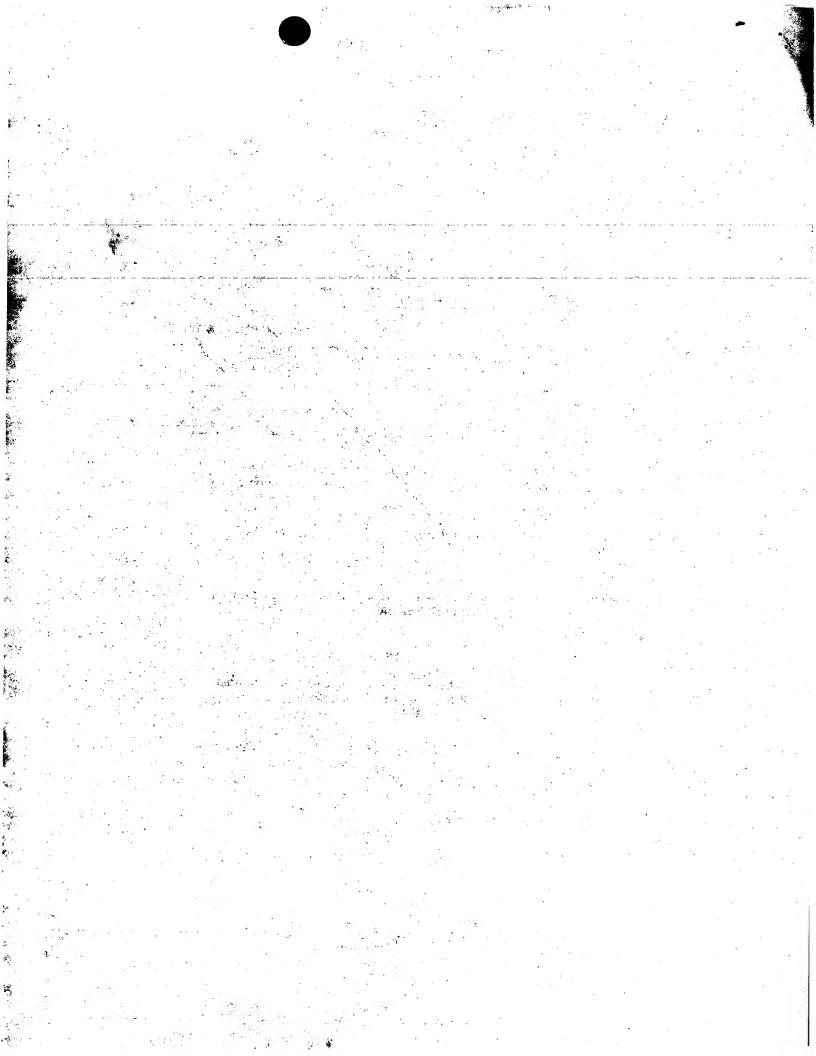
Authorized officer

Myers, J

European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d

Tel.+49 89 2399-8111





PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	1	See Notification of Transmittal of International
HMJ03045WO	FOR FURTHER ACTION	Preliminary Examination Report (Form PCT/IPEA/416)
International application No.	International filing date (day/mont	th/year) Priority date (day/month/year)
PCT/GB99/00064	08/01/1999	08/01/1998
International Patent Classification (IPC) or r C04B28/14	national classification and IPC	
Applicant MOBIL OIL COMPANY LIMITED e	t al.	
and is transmitted to the applicant 2. This REPORT consists of a total of the consists	according to Article 36. of 4 sheets, including this cover sided by ANNEXES, i.e. sheets of the asis for this report and/or sheets.	the description, claims and/or drawings which have containing rectifications made before this Authority
(see Rule 70.16 and Section These annexes consist of a total of	607 of the Administrative Instruct	tions under the PCT).
The second anniers of the second seco	,	
3. This report contains indications re	lating to the following items:	
II Priority		
	opinion with regard to novelty, in	nventive step and industrial applicability
IV Lack of unity of invent		
V ⊠ Reasoned statement		o novelty, inventive step or industrial applicability;
VI 🗆 Certain documents c		
VII 🖾 Certain defects in the	international application	
VIII Certain observations	on the international application	
Date of submission of the demand	Date of	f completion of this report
02/06/1999	·	1 0. 04. 00
Name and mailing address of the internation preliminary examining authority:	nal Authori	ized officer
European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 5236	Harbr	ron, J
		10 00 0000 0450

	The second secon				Wales .	a di managan di Andrea.
						•
			•			
a .			7.4	**	· · · · · · · · · · · · · · · · · · ·	
7				ā.		
1			·	· · ·		
		the state of the s	X			
			•		· ·	
1.34		The same of the sa	`		1	
					191	+3
	alan di seria a madalanda da mada da da 1966	THE A THE R. OF SEPTEMBER ASSESSMENT OF THE LAST CO. T. C. S. S.	and an and the second of the s	walion separate and the second second	a security of the security of	one of the second second second second
		+ 3				
F.				46		
N. in	4	in the second se				
					E ***	
					1	
						* · · · · · · · · · · · · · · · · · ·
					200	
				· d t		
				4500		
	10 ¹⁰ H					u. svi
4						90 1 1 V
				* *		
						*
4		*		* .		* ,
		60°				*
					*	Y .
\$						
ţ.		* =				
4						
1	a de la companya de l			*:	a di	
					F*+	
		* * * *				
		*	* * * * * * * * * * * * * * * * * * *	¥		
ž, o		•		and the second	• ***	
2		•				
Y			. · · · · ·		· -ce	•
3	•	i i		v.	ار مارچه	
ř.	*	* *				
	* .	•				. ()
			:		•	X-
9				•	* 2	. •
in t		14.	r y	* • • •		
*	•			*		,
	•	(4)	. 1		•	- Y -
*			\$			0
7	•.	47.		today of the		Y . •
		The state of the s	+ 1			(
	40 X	tion of the same o		at the state of th	**	

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB99/00064

 Basis of the 	e report
----------------------------------	----------

1. This report has been drawn on the basis of (substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.): Description, pages: as originally filed 1-13 Claims, No.: as originally filed 1-20 Drawings, sheets: 1/2-2/2 as originally filed 2. The amendments have resulted in the cancellation of: ☐ the description, pages: ☐ the claims. Nos.: sheets: the drawings, 3. This report has been established as if (some of) the amendments had not been made, since they have been established as if (some of) the amendments had not been made, since they have been established as if (some of) the amendments had not been made, since they have been established as if (some of) the amendments had not been made, since they have been established as if (some of) the amendments had not been made, since they have been established as if (some of) the amendments had not been made, since they have been established as if (some of) the amendments had not been made, since they have been established as if (some of) the amendments had not been made, since they have been established as if (some of) the amendments had not been made, since they have been established as if (some of) the amendment is a since they have been established as if (some of) the amendment is a since the considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

	·		*		in the state of th			· · · · · · · · · · · · · · · · · · ·
		* *			10.			
					. *			
N.						10 T		. S.
1								···,
						×		
IN.			No. 198		:	•		
¥	.,						•	1
			44 4 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1					
W.						. · X		
		* ***	70.			* .		
6						• .000		
*								7.
	بالمراج للاحم المجالة والأدارا	الاناس مستقد من (المناب المناسب المستقد). الراب الأناس المناسب		عاريشتان والمسوات		الماد والمواطنية الأراميني . الأرام	* * * * * * * * * * * * * * * * * * * *	gamenta (a) de seu de mente de la meste de la mest En la meste de
A.				¥,				
6								
<u> </u>		en e	بزيد وبفيد والمستدود		را عوارعيدا سا ساسيعدوسا	hata ay ang		<u> </u>
	A		*	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	11 - 11 - 1			
X					ie .	**		
						- 1 T	1	
37				No. To the			80	
				** = 3:				*
			A second		444			* .
3	*		1.400					
5 1 201		***	100			· · ·		
		55		. 940	Š.		• •	
		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		100			*	1 to
	- 18.			· :- · · · · · · · · · · · · · · · · · ·			Ţ.	
*						•		*
10	* * * * * * * * * * * * * * * * * * *	,						
Ac.				10.3				• •
N. BA	3000							
ħ.					**			
						*		· · · · · · · · · · · · · · · · · · ·
		**			• . • . • . •		. • •)	
			A STATE OF THE STA			•	£.	
			a negyan	ž				
*-	4	* * *		* •				
r _{en}					N.			
TW.			· •		4	,		
1.	*			36 1, 4	. •		,	
4			· · · · · ·					•
	•	*			•			
6								
7.4° 2.4								
		*				*	,	
	•				*.	. •		
1								
U		* * * * * * * * * * * * * * * * * * * *			*	*		
				<u>.</u>				
*. \$.	8 1	· ·	1	•		·		,
K ^{ris}		**************************************					. •	
				• • •		* •		
		7						7
6							,	
·			*					
*			* • • •			*	* ;*	•
						•		
rie.				,		-8-		
	· ·		*) · · ·				
and the second			- 3.,				. 4	
and the			*				12	- 5
			* 0.					•
	* - ·				•			
							· ·	
French Land			* *					
ii .	· · · · · · · · · · · · · · · · · · ·	· ·	* .	•				7

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB99/00064

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)

Yes:

Claims 1-20

No:

Claims

Inventive step (IS)

Yes:

Claims 1-20

1-20

No:

Claims

Industrial applicability (IA)

Yes:

Claims Claims

No:

2. Citations and explanations

see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

	8 •			* = -7	•			
2						· ·		
				•		•	* ****	
e.v.								
37	·							
-3				a •	7.7		' .	
1		*		,				
4			•		5-			
100				1.0		• 0		
2	· · · · · · · · · · · · · · · · · · ·							
· ·				. ,				•
			##(#) · ·		* * * * * * * * * * * * * * * * * * * *			
20	*							
4			, .	,				
			•	and the second second				
•	•					*		
90		1) N		.*		•		
-								
	and the second transport company common one and committee of the second transport of the second transp			الواسان الوادانية المستحدد والمشارد المستراد الم		and the second field and the second		
*					*.			*
200				<u>.</u>	2 7 8			
Sec				•				
200				i la la			*	
24						-		
-	Approximate and control of the contr	and the second second second	and the second	The state of the s	And the Annual of the Annual o			
			• 2.	*	Sin .	144	*	
100			Pol.			- 4 - 6		
1	A STATE OF THE STA			- X	***			
1						*		Α.
4				4 A F				
1								
		•		2		,		100
4				- Ac			·	
				• -				
				•				
				* * * * * * * * * * * * * * * * * * *				- 4
X4.			7 - 1	pm 30 1				
				•				Jan.
4. 3						ý		
	(a) W 102						•	
		•		**· -				- 3
				2.0				· 5
							•	
					•			45
		18					4	+
Co.			. 1 2 -			1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		A
20		•						· 🐔
27						4 7		
	$\frac{10}{3}$			•				
		4.				10.0		
					i i	• .		
all and								*
							•	
₹.	*							
F.	7					•		
1					, -			
							4.7	
					. 4			
					f			
2								
					·			
		•					· .	
8			•	· ·				
* .	•							
				_				
3		•	•	1				
			.* .	° ' •				
	· · · · · · · · · · · · · · · · · · ·						7.2	
1	v .			* -40		•		
× -							•	-
į.								
4.		10 - 20 - 10 - 10 - 10 - 10 - 10 - 10 - 10 -						
					1 2.			_
			•					
		· · · · · · ·		4.	· .	10	· * · · · · ·	
70.0							1 4.	•
P								
A. K.					•	The second		
							•	
	•							
A ₂					and the same		•	
	1					* *	· .	
154		* .						
		* 1						
7			8 4.			•		
j.			4				f	
4.								
	r en						ag · No	
2 37				**	13.7	i		
A 24			. ·			. 70		
No.			*					
5			·					
		17 15.	· 52"	- Pag	para de la companya		J+ 8 -	
1		* * * * * * * * * * * * * * * * * * * *		The Control of the Co		• *		
				and the same of th	14.5			

EXAMINATION REPORT - SEPARATE SHEET

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Reference is made to the following documents:

D1: US-A-5695553 D2: US-A-4315957

- 2. None of the cited prior art documents discloses all the features of any of the independent claims. In particular, the cloud point and foaming ability of the nonionic surfactant are not given in the prior art documents, so that these claims appear to be novel (Art. 33(2) PCT). However, it should be noted that the absence of certain properties of materials in a disclosure does not necessarily mean that said materials do not possess such characteristics.
- 3. Both of the prior art documents cited above disclose emulsifier systems which comprise nonionic surfactants and sulphated anionic dispersants. D1 discloses a poly(phenolate carboxylate) resin emulsifier with a lignosulfonic acid dispersant (column 3, lines 40-59) and D2 also teaches the use of nonionic emulsifiers which may be combined with sulphated anionic emulsifiers (column 3, lines 34-45). Neither of the prior art documents disclose nor suggest an emulsifier system having the properties given in the characterising portion of the independent claims. Furthermore, there is no reference in either document which would lead the skilled man to combine the teachings of one with that of the other. The application is considered to involve an inventive step (Art. 33(3) PCT).

Re Item VII

Certain defects in the international application

1. Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents D1 and D2 is not mentioned in the description, nor are these documents identified therein.

4.4		
		♥ 3.
		· 'Y
E		
3		
2	5 M 3	
4.		
5		
ř		
Š4		
		2 40 "10"
-		
(a)		
Ŷ		*
> *,		
		and the second
andrew .	المنافع والمناف فأناف المنافع والمارية والأراكية فيناه والمرافع المنطقين المنافعين فالمنافع والمنافع والمنافع والمنافع	
2,		•
12		
8 4	The state of the s	
2		•
	그리고 그는 그는 그를 가는 것이 되는 사람이 있는 그 없는 지원에 가장 그 때문에 그를 가장 하는 것이 없다.	
100		
	그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그 그	
•	그 그 그 그 그는	*
Ž.		
2	그는 그 보고 있는데 바람이는 사람이 바쁜 취임에 어느 것 같은 사람이 되는 사람이 되는 사람들이 되는 것 같은 그 모양이다.	
	· 사용	
2	그는 그는 그는 사람이 하는 회사들의 사람들이 되는 그리를 함께 하셨다. 그는 그는 그는 가는 그를 모르는 것이 그리는 것들이 되었다.	· · · · · · · · · · · · · · · · · · ·
	그는 이 그리고 한 경험을 하는 것들은 그렇게 그렇게 하지만 하는 것이 하셨다면 하는 것이 되었다. 그렇게 되었다면 하는 것이다.	
*	그리고 그리고 살아왔다. 그 그 그는 요리를 가장하는 그 그들은 그들은 사람들이 얼마나 나를 가장 하는 것이 되었다. 그는 것이 없는 그는 것이 없는 것이다.	***
L	그는 그 그 그 그는 그 이렇게 되는 것을 하는 사람들이 되는 것이 되는 것이 되는 것이 되었다. 그 그 가는 사람들이 가득하게 되었다면 되었다면 그렇게 되었다.	
reserve .	그리지 그 그 그는 아들께서는 그 그는 것이 그는 그들은 사람들이 얼마를 하는 것이 되었다.	107
1	어린 하는 사람들은 사람들이 가장 아니는 사람들이 가장 하는 것이 되었다. 그 사람들은 사람들이 가장 아니는 사람들이 되었다. 그 사람들이 되었다.	
27		•
2		
*		
Project Comments	그는 그 그는 그는 그는 이렇게 되는 것이 되는 것이 되었다. 그는 그는 이번 사람들은 그는 사람들이 되었다. 그 사람들은 그는 사람들은 그는 사람들은 그는 그를 살아보는 것이다.	
d it	어린 사람들은 사람들이 얼마나왔다면 하고 있다면 하고 있는 것이 되었다. 그 사람들은 그들은 그렇게 하는 것이 되었다면 하는 것이 없다.	
**	그는 그리고 있다면 그는 사람들이 나는 사람들이 그 아무리는 그는 그리면 생활을 하지만 하는데 모든 가장이 되었다. 그는 그 모든 사람들은 그는 그를 다 되었다.	*
5		• •
	그러면 그 그 그는 그를 하는 아무셨다면요. 그렇게 하면 하는데 그 말했다면 그는데 하는데 그리고 있다고 말하는데 그렇게 되었다.	
100		
n's	그 하는 그는 그리는 걸려면 그리고 바다 바다를 하는 것이 되었다. 그는 그는 그는 그를 되었다.	
e se		
*	그는 그들이 그는 그를 가장 가는 한 것 같다. 그렇게 되는 바다를 가장 되었습니다. 그는 그를 가장 하는 그는 그를 모르는 그를 되었습니다.	
	그는 그리는 요즘, 이 성진 내용, 요즘 한 경인 아이 성격 사용적인 사람들이 아니는 아이는 아이들이 되는 것이다.	
	그는 그 사람들 선생님들이 하는 것이 없는 것이 되는 경험에 가면 가장 살이 되었다.	
A Pa		
2 A		
3		
The second secon		
The second secon		
The second secon		
The state of the s		
The second secon		
The state of the s		
The state of the s		
The second secon		
The second secon		
And the second s		
The state of the s		

WORLD INTELLECTUAL PROPERTY ORGANIZATION International Burcau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6: C04B 28/14, C08L 91/06 // (C04B 28/14, 24:08, 24:22, 24:32, 24:36), 111:27

(11) International Publication Number:

WO 99/35103

(43) International Publication Date:

15 July 1999 (15.07.99)

(21) International Application Number:

PCT/GB99/00064

A1

(22) International Filing Date:

8 January 1999 (08.01.99)

(30) Priority Data:

9800368.4

8 January 1998 (08.01.98)

(71) Applicant (for all designated States except US): MOBIL OIL COMPANY LIMITED [GB/GB]; Mobil House, 500-600 Witan Gate, Central Milton Keynes, Buckinghamshire MK9 1ES (GB).

(72) Inventor; and

(75) Inventor/Applicant (for US only): BOELEE, Scotia [NZ/GB]; 105 Forest Road, Tunbridge Wells, Kent TN2 5BG (GB).

(74) Agent: GILL JENNINGS & EVERY; Broadgate House, 7 Eldon Street, London EC2M 7LH (GB).

(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR. BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published

With international search report.

Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: GYPSUM PRODUCT

(57) Abstract

A wax emulsion comprising an emulsifying system containing a sulphated anionic surfactant and a non ionic surfactant having high water solubility (cloud point) and high foaming ability is added to a gypsum slurry to improve the moisture resistance of gypsum board. The wax is a mixture of a petroleum derived hydrocarbon wax and a montan wax.

ļ	<i>]</i>		ng transmission of the state o	A			
1	$\int_{\mathbb{R}^2}$				1 8	· H	
1	*				. 0		
I_{s}							
Y				·san en en			
		*.	Ų.		*		
V.		* *	*·		v .		
-			in the second se		· · ·		
	d					en en	
	•	1.1	T .		***		
		0.			*		•
			1-			to:	
28		**			•		•
0		egi kangamen dan Kilabara. Kangamen			a		
24							PX
			7.1				*
eli, e i					and the second seco		
F1941		3	** ***			. (a)	
7 J		•					
				X X X X X X	0 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		
						7	
(e)				8 · *			
¥							
<u>.</u> E		*				La re-	
		()		17			
					0.7	H4.	
	- **	- ±		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1			
					- ×		i di
		. X			* *		
Ú)		* .	***	** 4	g (8)		
ż		*.*				171	
			*	_			
10						0 , = -	
			ting the second of the second				
					*		
i at	•						
d.		*	4 40			90 Y	
4.	٠.		A Comment of the second		-		
	٠.,					* .	
						*	
				es es			
						X1.50	, p***
è.	2.						
					1		
71 -							
			* * * * * * * * * * * * * * * * * * * *			. *	
			-				. **
							· · · · · · · · · · · · · · · · · · ·
					*		
		- 0,			, , , , , ,	* *	
				0 2 8		* ** ** ** ** ** ** ** ** ** ** ** ** *	K'j
			*				
ł .					* **		i j
f.,				· · · · · · · · · · · · · · · · · · ·	,).c		ý.
'A'É			<u>`</u> , •			*	, Å
			*		,	7 '	
i,			÷		•	•	
F			*	110	* •		
			,	*			
Ny Li		21 V ₂		- 4	⊕ 		
Br.		• •		" N "		W. '	
9		e and	* * * * * * * * * * * * * * * * * * * *			- 1 7 m	
100							
		4.0	Market 1			1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	1410 m

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain		LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland		LT	Lithuania	SK	Slovakia
AT	Austria	FR	Prance	,	_rn	Luxembourg	SN	Scnegal
·AU	Australia	GA	Gabon		LV	Latvia	SZ	Swaziland
AZ.	Azerbaijan	GB	United Kingdom		MC	Monaco	TD	Chad
PA.	Bosnia and Herzegovina	GE	Georgia		MD	Republic of Moldova	TG	Togo
ВВ	Barbados	GH	Ghana		MG	Madagascar	TJ	Tajikistan
BE.	Belgium	GN	Guinea		MK	The former Yugoslav	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	. ,		Republic of Macedonia	TR	Turkey
BG	Bulgaria	HU	Hungary		ML	Mali	TT	Trinidad and Tobago
BI	Benin	IE	Ireland		MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel		MR	Mauritania	UG	Uganda
BY	Belanis	IS	Iceland		MW	Malawi	US	United States of America
CA	Canada	ľľ	Italy	_	MX	Mexico	UŽ	Uzbekistan
CF	Central African Republic	JP	Japan	•	NB	Niger	VN	Viet Nam
CG	Congo	KE	Kenya		NL	Netherlands	Ϋ́Ū	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan		NO	Norway	zw	Zimbabwe
CI.	Côte d'Ivoire	KP	Democratic People's		NZ	New Zealand		2
CM	Cameroon		Republic of Korea		PL	Poland		
CN	China	KR .	Republic of Korea		PT	Portugal		
CU	Cuba	KZ	Kazakstan	•	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia		RU	Russian Pederation		
DE	Germany	LI	Liechtenstein		SD	Sudan		
DK	Denmark	LK	Sri Lanka		SE	Sweden		
EE	Estonia	LR	Liberia		SG	Singapore		

716.4		-= <u>, </u>		. == 		-	= =
	•			÷ ,	A	•	*
		•			4		
		-		•		•	•1
	•						
	٠.						
			•				·
	•			*			
		***		<i>i</i> ,	· ·		
	* , A	-0			¥		18
	•	*	1				
		0.0			, ,	*r *	
	•			•			\$
							· · · · · · · · · · · · · · · · · · ·
بالسفاد فيا يوال وسفور وبراس للشيسويون الرا	الله ويا المطالبيسية السناء فالطائف أفاله ليواريو	مرياع قب تنگ لدن به ايد شنشد دا چاريد و	سالفادو، ساد درسمارسسا	ومر فهنتم تعسست	بالروائد ساء شباطأ السابطان سيسف		and the same state of the same
	*				.00		*
	Ť.						
48	8				· · · · · · · · · · · · · · · · · · ·		
		*		· *	9 '	•	
	*	:	+	, (<u>)</u>)			
	· (4) · · · · · · · · · · · · · · · · · · ·		*	· · · · · · · · · · · · · · · · · · ·			
*	* 1				*.		
	1.						
	× .	*			*		· .5
		*			· ·	3.	4
	, , , , , , , , , , , , , , , , , , ,			Ža,	O		10
			* .		, "		
X-			No.				
	0	• •					*
			, , , , , , , , , , , , , , , , , , ,			1	*
		• • •				I	
			*		,		
						•	
				,		· Ž	•
			3.				· Y · · · · · ·
	* 4						
				X			•
	* .						
	*		10 mg - 10 mg	eX-		**	
			40 *		*	-()-	* T
8				:-	•		
•			. 8.	•			
.			180				.0
			0		`f.	- 170	
			*		ė .		
			•	•	•		
	χ.						
*,		1					
		* * .	-X-		·	•	
				*	•		
		•					
The second secon						• •	
	\mathcal{A}_{θ}	*				**	
	•			*	•		

GYPSUM PRODUCT

This invention relates to a gypsum product and to a process for its manufacture. More particularly, this invention relates to a foamed gypsum product of improved water resistance and/or reduced density and to a process, preferably to a continuous process, for its manufacture.

5

10

15

20

25

Gypsum board (or plaster board or wallboard) is used extensively in the construction industry. It typically comprises a substantially flat core of set gypsum on either side of which a liner may be adhered. A liner typically comprises paper. The core may be reinforced; for example, reinforced with glass fibres.

Gypsum products (or Plaster of Paris or plaster products) are produced by mixing anhydrous calcium sulphate 🔧 or calcium sulphate hemihydrate with water, and permitting the mixture to set thereby producing calcium sulphate dihydrate. Often the slurry is foamed by incorporating a preformed solution of foaming agent in water (a surface active material) before adding to the mould means. pervasive problem with gypsum products, however, is that calcium sulphate dihydrate absorbs water and this reduces the strength of the gypsum product. Because of this, plaster board (for example) is required, at least in uses where a relatively high humidity is anticipated (for example, kitchens or bathrooms) to be substantially moisture resistant and this requires the presence of a hydrophobing agent. ("Hydrophobing" is a term used in the art to denote a method of preventing, or reducing water absorption).

30 Silicone oil has previously been used as a hydrophobing agent for gypsum products. It is, however, expensive and in relatively short supply. It also has

order of the company	<i>:</i>	· · · · · · · · · · · · · · · · · · ·
	•	
and the second of the second o	· · · · · · · · · · · · · · · · · · ·	-
	•	, P
		- 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1 - 1
	•	*
		00
	,	
		•
	والمنطف سال ملازه فروا بحرسه	المستسود بماء والمرسمونية والتنام
		- 100
	- 2 ²² - 2	
	and and all a part of present	The state of the second of the
	in Lear	
		*
	. 4	
	0.	
	` ` ` ` ` ` ` ` ` ` ` ` ` ` ` ` ` ` ` `	

	, a**	28° - * n - 4
	· .	
	*	
	•	
	J	
		•
		•
	\	
	•	
		*
		* * * * * * * * * * * * * * * * * * * *
	. *	
	. •	,
\cdot		•

difficulty in providing a moisture resistance of less than 5 wt % water absorption in the test hereinafter mentioned.

In US-A-5437722 an aqueous emulsion comprising a hydrocarbon wax, a montan wax and emulsifier/stabiliser system and also including a polyvinyl alcohol, is used to render gypsum products water resistant. The emulsifier system may include non-ionic or anionic surfactant and alkali. Examples of non ionic surfactants are alkylphenoxypoly(ethyleneoxy) ethanols, sorbitan fatty acid esters and polyoxyethylene sorbitan fatty acid esters. Examples of anionic surfactants are saponified fatty acids.

10

15

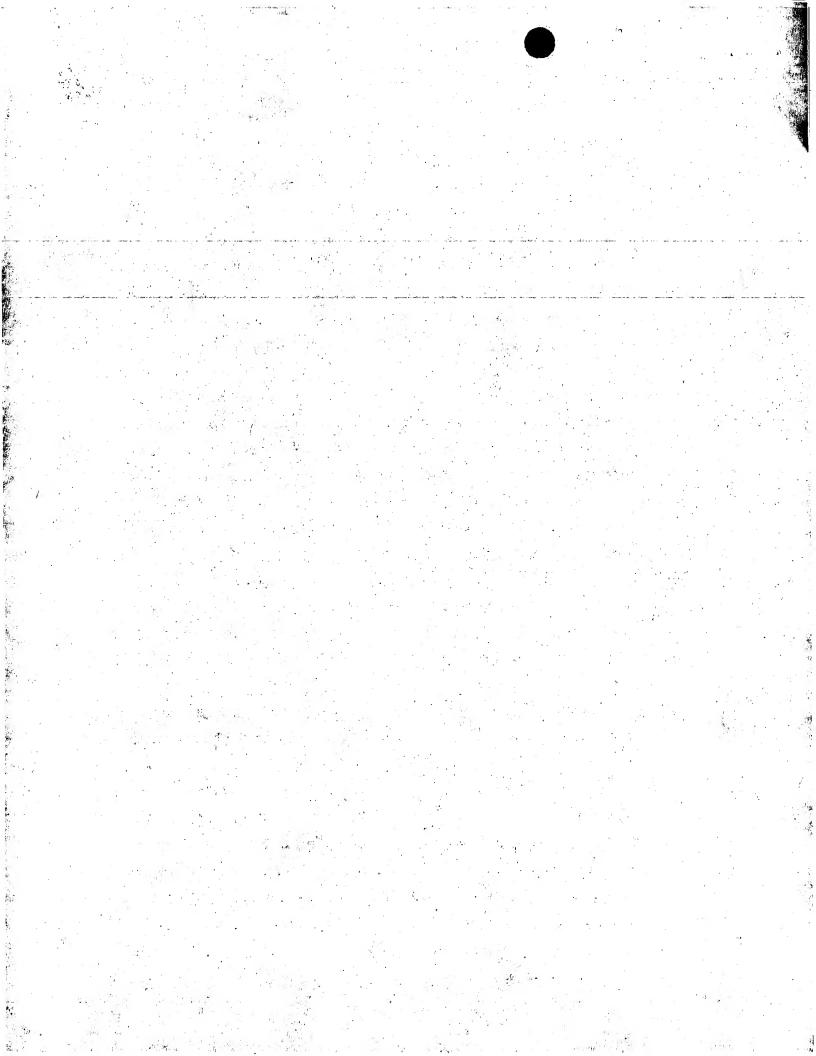
20

25

30

In our copending application number PCT/GB97/02366, unpublished at the priority date of the present invention, we describe a foamed gypsum product which is hydrophobed by incorporation of an aqueous emulsion comprising a hydrocarbon wax, a montan wax and a colloid stabilised emulsifier system. The preferred colloid stabilised emulsifier system comprises either organic or, more preferably, inorganic colloidal materials. One example is, a montmorillonite clay based system in combination with a sodium naphthalene sulphonate.

In the above mentioned PCT application, a comparative example uses an emulsifier system based on a combination of a nonionic surfactant, which was not specifically defined, with an anionic surfactant again, not specifically defined. The emulsifier system used in that comparative example was in fact a combination of an alkyl phenyl ethoxylate with a soap-type anionic surfactant. The worked example using that emulsifier system did not work. In fact it is now believed that the anionic surfactant caused collapse of the foam or that, upon addition to a slurry formed using



relatively hard water, the anionic surfactant was precipitated out of the system by the hardness ions.

The above mentioned PCT application also discloses a comparative example using a nonionic surfactant based emulsifier system including no anionic emulsifier. Whilst this gave some improvement over the mixed system, it was found that using a high enough level of emulsion in the gypsum product to achieve adequate density and/or water adsorption figures lead to over-wetting of paper used for the gypsum product and delamination during production.

5

10

15

20

25

30

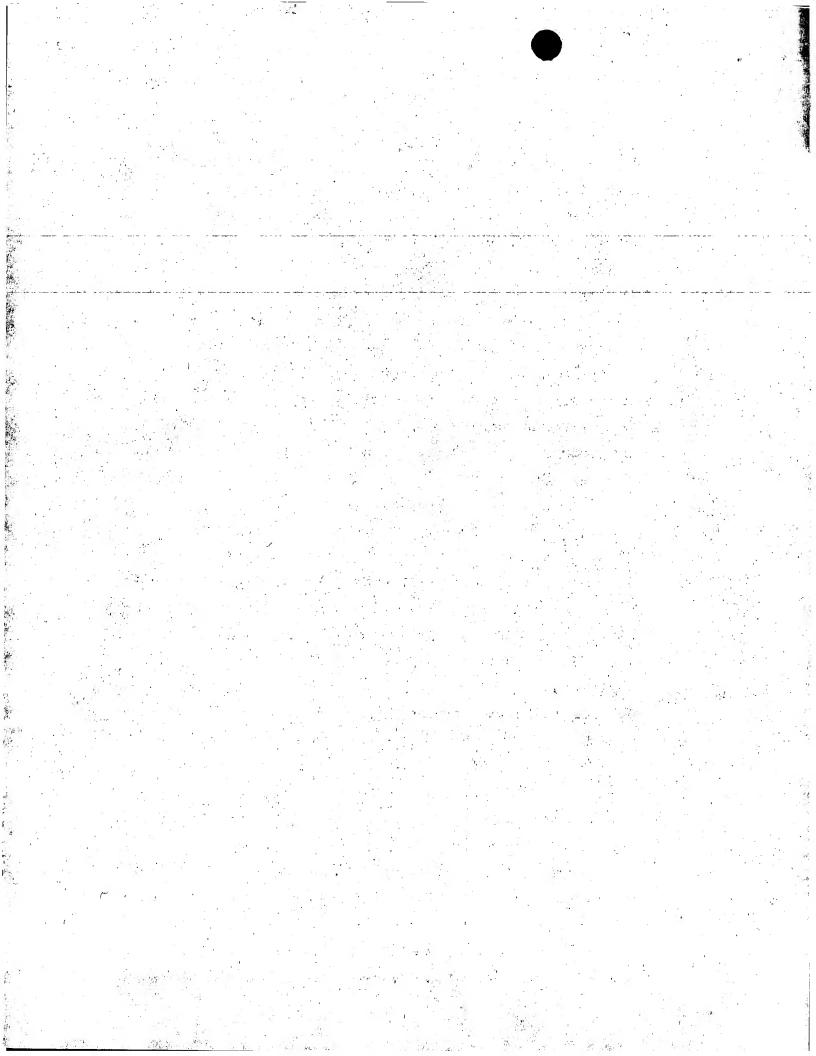
In the present invention there is a provided a process for producing a foamed gypsum product involving the following steps:

- a) a slurry of gypsum is formed in water
- b) the slurry is introduced to mould means and allowed to hydrate,

in which a hydrophobing agent comprising an emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system is added to the slurry before introduction into the mould means, and is characterised in that the emulsifier system comprises:

- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point (in saline per DIN 53917) of at least 50; and
- ii) an anionic dispersing agent which is a sulphated compound.

Preferably the anionic dispersant is a so called acid stable compound, that is the compound is ionised over a wide range of pH's including acidic pH. The acid stability can be judged by the pK_a of the conjugate acid, which should preferably be less than 4, more preferably less than



4

3, for instance 2 or less. The anionic dispersant is generally a sulphate or a sulphonate.

A suitable class of anionic dispersants are sulphated naphthalene/formaldehyde condensates, for instance having molecular weight in the range 6000 to 40000. These compounds are also known as naphthalene sulphonates. Other aryl sulphonates may also be used. The anionic dispersant is generally used in the emulsion in the form of its sodium salt. Alternatively potassium, ammonium, or even divalent metal salts such as calcium or magnesium, may be used. Suitable compounds are available from BASF AG under the trade name Tamol (trademark).

The nonionic surfactant must be relatively water soluble. The water solubility of non ionic surfactants can be determined by standard test method DIN 53917 in saline. The component should have a cloud point of at least 50, for instance more than 60, up to around 100, for instance approximately 75.

15

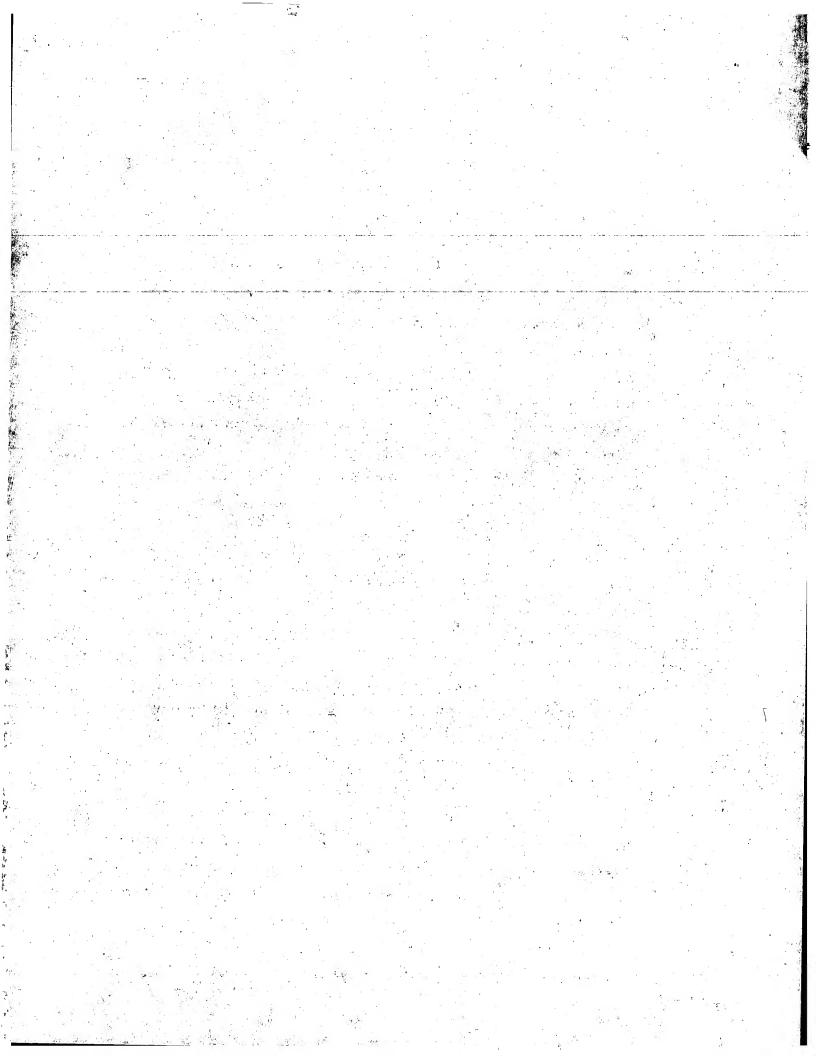
20

25

30

We have found that the nonionic surfactant giving optimum performance is one which has a high foaming ability. Foaming ability can be measured by standard test methods DIN 53902. For instance the test should be carried out according to the method given in sheet 1 of that standard test method, at 40°C, with the surfactant being used in a concentration of 2 g/l in water containing 1.8 mmol Ca ions/l, the duration of the test being 30 seconds. The foaming ability should be at least 300, more preferably at least 500, for instance up to 750. Nonionic surfactants with foaming ability using the above mentioned test method of around 600 are available.

The nonionic surfactant is generally an ethoxylated higher alkyl, alkenyl, alkanoyl or alkenoyl compound.



5

Alternatively ethoxylated aryl compounds may be used, for instance ethoxylated alkyl phenol derivatives. Preferably the compound is a C_{8-18} -alkanol ethoxylated with 3 to 30 equivalents of ethylene oxide, for instance a C_{12-18} -alkanol ethoxylated with 10 to 20 equivalents of ethylene oxide. Suitable compounds are available under the trade name Lutensol (trademark) from BASF AG. It may alternatively be possible to use polyglycosylated alkyl, alkenyl, alkanoyl, alkenoyl and aryl compounds for instance alkyl polyglucosides.

The use of an acid stable anionic dispersant, it is believed, avoids the addition of the emulsion resulting in the collapse of the foam in the gypsum slurry. Accordingly the density of the product is optimised. The use of the anionic dispersant in combination with non ionic surfactant avoids the use of such high concentrations of nonionic dispersant in the gypsum slurry which can lead to overwetting of paper used in the gypsum product and delamination during production. The preferred surfactant makes the emulsion suitable for use with gypsum slurries made up in hard water, for instance water having hardness value of at least 100 ppm Ca²⁺ even more than 150 ppm Ca²⁺¹, for instance at least 200 ppm Ca²⁺.

15

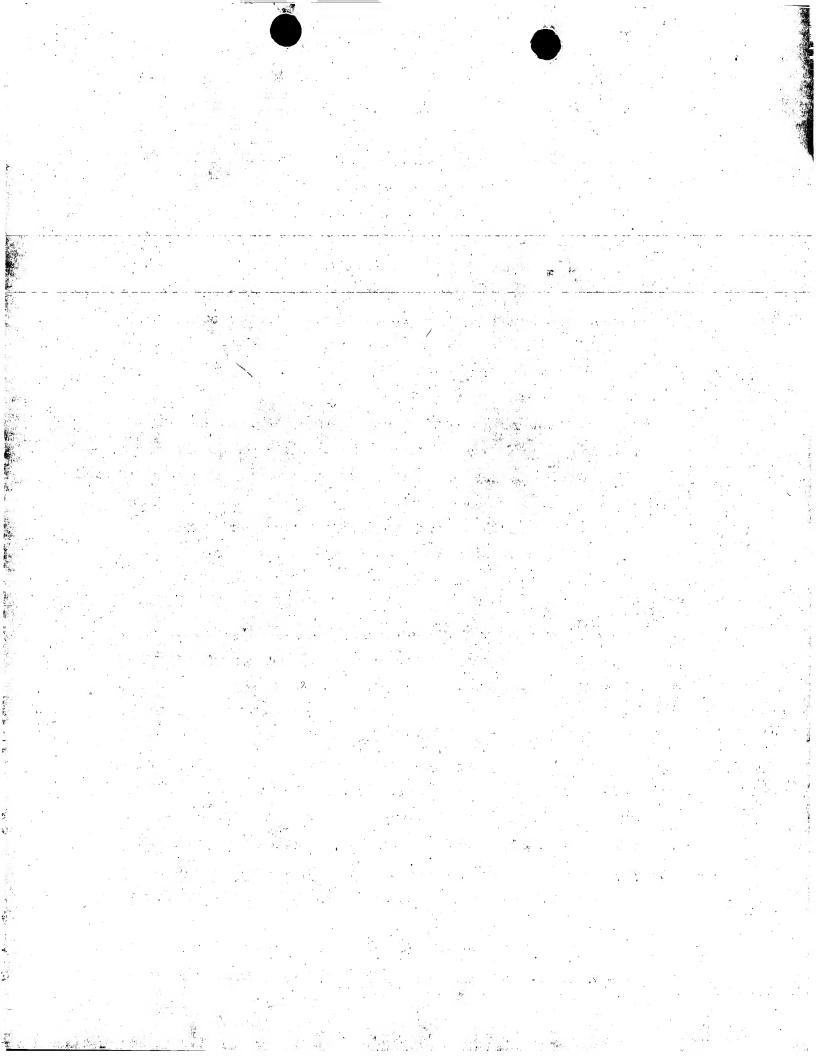
20

25

30

The use of the high foaming non ionic surfactant is believed also to contribute to optimisation of the gypsum slurry foam and the density of the final product. The preferred combination of surfactants in the emulsifier—system allows high levels of wax to be incorporated into the final product for optimum hydrophobing of the gypsum product.

The emulsifier system has process advantages also in production of non-foamed products, for instance, other



10

products made on continuous lines such as fire resistance The system provides good compatibility with the equipment

petroleum-derived hydrocarbon The wax preferably one with a high melting point and a low oil content. A preferred such wax is a paraffin wax, such as fully refined paraffin wax. Fully refined paraffin waxes are generally obtained from highly paraffinic refinery streams such as those obtained from the solvent dewaxing of distillates and other lube fractions. The product is further typically characterised as follows:

CHARACTERISTICS	TEST METHOD	SPECIFICATION		
· · · · · · · · · · · · · · · · · · ·		MIM	MAX	
Congealing Point (°C)	ASTM D938	55	69	
Oil in Wax (%)	ASTM D721		1	
Penetration at 25°C (mm/10)	ASTM D1321	10	20	
Penetration at 50°C (mm/10)	ASTM D1321		80	
Viscosity (cSt @ 100°C)	ASTM D445	3	7	

25

30

An example of a fully refined paraffin wax which has been found to be entirely satisfactory, and which satisfies the above specification, is MOBILWAX 135 (derived from the 150 SPN stream) as supplied by Mobil Oil Company Limited: MOBILWAX 145 or 150 (derived from the 300 or 450 SPN stream) are also suitable. While these waxes hydrofinished to give a white colour and good odour, unfinished wax (which differs only in colour and odour) is also suitable for use in accordance with the invention.

	•				38		•	,			<i>x</i> •	•			٠.	
			1.		a a	ļ.	•							•		
	- 1	•		,												đ
			141									*	•	•		
			.*									• .				130
			,													
ul.				, f												
					A second		, in				, in					
4		* 0			1 00					•		. *				
Ann.														1		
ř.																-
ų.			5 mm m				21 A-40 - 404			+		English on which remains				
2"	4			•						. *	*					
						* .	54									
	وتوال والمسأ	ست خیب عا	ر بوشاوست لد م		بيعير الماسية منهد	/ <u></u>		eggin en en en en	بسيد الخاطية	والداعة بجدادت لحسمها	-	بالما الماسية عسوالله	ogiana della media della d		اقا باز اقتامید دارد را اقتامید دارد را	·,
500 - 2 1				- + 1	1	**		• •		· · · · · · · · · · · · · · · · · · ·					* • •	
Ý.			, * ' *'	3												
			·		L	1		in the		*				X		
										* .	=		- * · · · · · · · · · · · · · · · · · ·		fig. s	
100			71				X			*						
		ş.		2		1.8		3. *	equ ¹		1				0- 11	9
		200						17			v (f	** *** *** *** *** *** *** *** *** ***	i		- ta	
			Н												- 3	. 1)
	*				NIE					ē				,		7.0
k.		A							, -	3-1						
			ay to						-10-	- jan -				,		
7	1								x - 1 - 1							. '
						5	m									
1.7								* `				. I ta				
Ċ.				-11					٠.							1
, i				H 1	· .	*			* . ' '			7 =				
				· · · · ·		2.1						11.	*			, .
*	**			*	over the part		4,700	···		•						, -
							:	(x)		1.19	بعراث وو		1,85			· ·
										J.		***	0.0			
								1	• . •				* *			>
1			'							-						
8										- '	**				1- 1	100
						· · · ·	207	4.1	i .	fra.		•		Ů,	•	
	•				, J ₂ ,			- 0				= ,,				
· ·				. 1.				•				*			,	•
					0			· .								
h.				4 M	4										-	
							. 1 . 1,		+2						· ·	
	F. 8		*,	ė				: ند	*		•					
							-	*						0	7	
Ny.		**		40												100 - 10
· #					74. w	- 4 - 38					3:				. *	
						$\mathcal{A}_{i,\alpha}^{f}$		77	* **				,			
					34.				1				* .			
				- FR	÷ .				•			0. 2			. *	
				10.			-			*	9.			0.4	,	
<u>.</u>			2						•	· #*		4		* * * * * * * * * * * * * * * * * * * *		
			**		1							1		.,		·
				Carrier Control	**	3.		٠								8
(-X.Y.			(4.0)		P		X	-	-4		in the second				
	100	7	5 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	1 TO 1		11 mm		10.00			16 3	e c.				

7

The petroleum-derived hydrocarbon wax a) suitably comprises from 20 to 40 wt % of the aqueous emulsion, preferably from 25 to 35 wt % of the aqueous emulsion.

The montan wax or lignite wax b) is another wax with a high melting point. It is preferably used in crude (or raw) form. Such a product is typically characterised as follows:

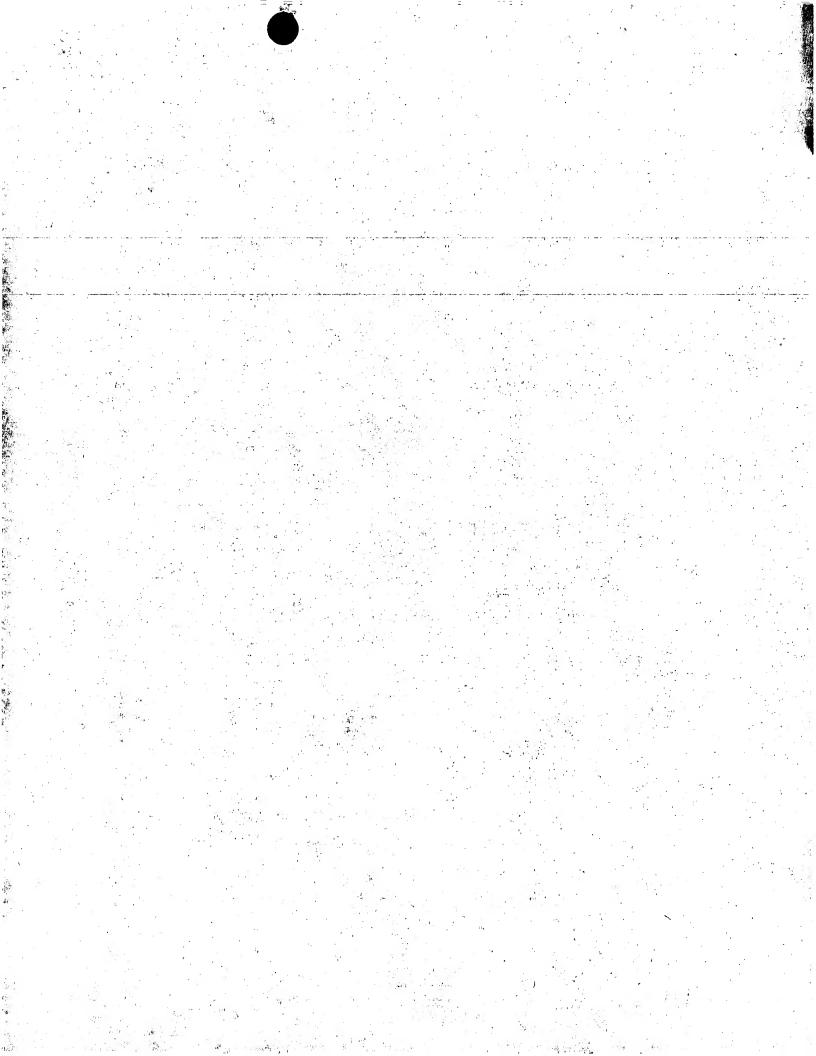
				1	
CHARACTERISTICS TEST SPECIFICATION METHOD		CATION	PREFERRED SPECIFIC- ATION		
		MIN	MAX	MIN	XAM
Congealing Point (°C)	ASTM D938	67	8.0	75	80
Acid Value (mg KOH/g)	ASTM D1980	10	37	10	20
Saponification Value (mgKOH/g)	ASTM D1962	35	100	65	90
Ash Content (% wt)	ASTM D482		1		1.0
Density at 20°C (g/cm³)	ASTM D1298	0.95	1.04	0.95	1.04
Viscosity (cSt at 90°C)	ASTM D445	20	400	150	400
Viscosity (cSt at 100°C)	ASTM D445	20	200	. 60	150

25.

30

The montan wax b) suitably comprises from 10 to 20 wt % of the aqueous emulsion, preferably from 11 to 15 wt % of the aqueous emulsion.

The emulsifier system (i.e. the total of two or more components of a multi-component system) is suitably present in an amount from 0.5 to 6 wt %, preferably 12 to 5 wt %, more preferably 1.5 to 4% of the aqueous emulsion. The



8

ratio of the non-ionic and anionic components is preferably in the range 5:1 to 1:5, more preferably 3:1 to 1:3, most preferably 2:1 to 1:2.

In the invention the slurry in water, preferably contains 100 parts by weight of gypsum and from 0.5 to 10, preferably from 1 to 5 % by weight of an emulsion as herein defined. The slurry suitably contains 50-60 weight % gypsum and 40-50 weight % water, preferably about 55% gypsum. An accelerator is usually added, for instance a slurry mix from a previous batch.

The slurry preferably contains a foaming agent. Preferably the product is a paper lined board and the process thus preferably includes a step of foaming, usually involving formation of a pre-formed foam by vigorous stirring of the foaming agent in water, followed by mixing the prefoam into preformed gypsum slurry containing the emulsion.

15

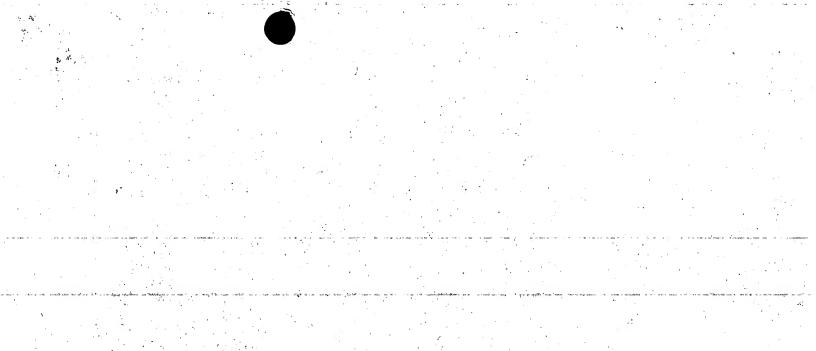
20

25

30

This invention also provides a water-resistant gypsum product which comprises the set composition. Whilst the product may be an unlined board, the invention is particularly applicable to a product which comprises a core product of a set such composition sandwiched between a pair of liners usually paper liners. Another suitable product is fire resistant board which has a glass fibre scrim embedded in each surface of the gypsum board, which is generally unfoamed. The invention includes also the emulsion itself and the process for making it.

The invention further provides a process for the preparation of a water-resistant gypsum board product, which process comprises forming a mixture which is a slurry in water containing 100 parts by weight of gypsum and from 0.5 to 5 parts by weight of an emulsion according to the



•

5

10

15

20

25

9

invention; forming a layer of the mixture in a mould means and drying the layer of gypsum mixture, while permitting hydration of the gypsum, for form a board product. Preferably the process is continuous. The process may be for forming tiles or blocks or boards. Blocks may be formed in moulds from which they are removed when set. Tiles or boards may be formed by spreading a layer of the gypsum mixture on a first planar substrate, a second planar substrate is positioned over the layer to form an assembly, and the mixture is allowed to set in the assembly. gasket may be provided between the planar substrates. Where the product is a lined board, the first and second planar substrates are constituted by liner, for instance paper, usually supported in a mould. Where the product is to be unlined, the planar substrates are removed when the product is set. Where the product is a block, it is usually removed from a mould before the mixture is completely set, but when it is hard enough to handle. Where the product is a fire resistant board a fibreglass. scrim is embedded in each side of the slurry in the mould means by feeding to each side of the poured (unfoamed) slurry before the nip forming a dam in an apparatus similar to that shown in Figures 3 and 4 below.

This invention includes the use of an aqueous emulsion of the invention to furnish a gypsum product with water resistance and the use of an aqueous emulsion of the invention to aid foaming of a gypsum slurry, for instance to reduce the density of the set foamed gypsum product.

Figure 1 shows the water absorption results of the product of the comparative example.

Figure 2 shows the water absorption results of the product of the example of the invention.

 $x = \frac{1}{2} i \mathbf{k} \cdot \mathbf{r}^{T}.$

10

Figures 3 and 4 are a schematic representations of a foaming station for lined gypsum board.

The following Example illustrate the invention.

EXAMPLE

5

10

15

25

30

First the wax phase was prepared by dissolving 12 wt % crude montan wax (Crude Montan Wax supplied by Schuemann Sabol GmbH) in 30 wt % of fully refined paraffin wax (MOBILWAX 135 supplied by Mobil Oil Company Limited) at a suitable raised temperature. 1% by weight sodium naphthalene sulphonate (a sulphated naphthalene formaldehyde condensate having a molecular weight of about 6000 to 40000) from the TAMOL (trademark) range supplied by BASF was added to water to form the aqueous phase and stirred for a period. 0.45% Non ionic surfactant (added as a 90% aqueous solution) (a C, alkanol - 12 mole ethoxylate available as Lutensol TO12 series) was then added to the aqueous phase and stirred for a period. The temperature of the wax melt was lowered to 100°C and the wax phase was next added to the aqueous phase heated to a suitable 20 temperature, with stirring for a suitable period to form a The pre-emulsion still at a raised pre-emulsion. temperature was next recycled through a homogeniser, with no impressed pressure, for a full pass. Gradually, the pressure was increased to a value in the range 20-25 MPa (220 bar) and the emulsion recycled for a further pass to form an aqueous emulsion in accordance with the invention.

The emulsion was then tested for its performance in the production of a gypsum product. A conventional foaming agent was mixed with vigorous stirring with a suitable quantity water to generate a foam mixture. A gypsum slurry mix was prepared by adding a predetermined amount (1.4, 1.6 or 1.8% by weight based on the amount of gypsum) of wax

-			प्रचालकः ≱रू		₹ • •		0:		
				* * * * * * * * * * * * * * * * * * *			• • • • • • • • • • • • • • • • • • • •	¥1.	
			,	*					
PT.		3. 3. 3. 3. 3. 3. 3. 3. 3. 3. 3. 3. 3. 3				•			

i.					*				
		4. 12 ×							~ 1
									*
								: 	· ; *
	*								
		ريده د در د	Aykan akan dan dan dan katan manan katan dan dan katan dan dan dan dan dan dan dan dan dan d	يود د پېرونوس بغير د د دستهرو واستيد	an intermediate and the contract of the contra	سيواهد والمستداد المالية	- Hanneston State of the State	ئىقاسىد چاندادىد	
1								•	(1) (1)
5	*			70					
£		·							-
Č.									
3			144			* * *			
					Mary .		2-	e .	
	*	112.77				1 and 1	\$1. (A. 1)		
47		Vac.							
		**					e e	- 2 A.	
									· · · · · · · · ·
		1		*			i		
	6				**			•	
		1.4						* ,	
		· A							
24				13 mg - 23 mg 1 mg - 21 mg	***	, , , , , , , , , , , , , , , , , , ,			
(P) (P)(A)				e y e (tree			e ••*		
	• , (2 a	. 1.	

		= = 0 0		e to the second					
3		100 pt	i de la companya de l			V			
		* 4						·.	
Ä.	. *			.0		× 4.			- 4
*	•				a 7 - 7 ×	,			
				(1)			· ·		
		•							.*
2					*	an .			
9 7.		× 1, 3	erentis. Notas sur sur sur sur sur sur sur sur sur su	37 - 3	^k * ;		W		
L.		,			**		u u	. 20	
is.	· · · · · · · · · · · · · · · · · · ·	S.			21.10	239 ·	e de la companya de l		

15

20

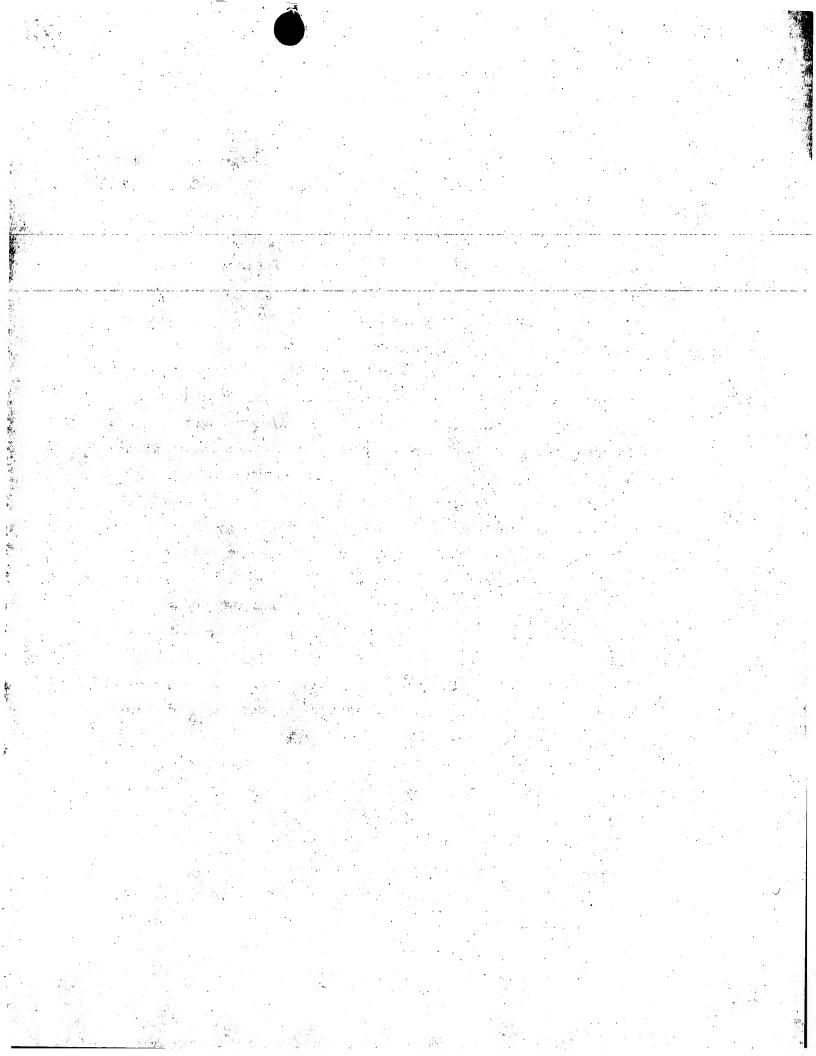
25

30

emulsion (according to the invention or comparative) to around 40 parts by weight water along with predetermined amounts of a wetting agent, starch and an accelerator in a total amount of 0.38 parts by weight. To this around 58 5 parts by weight gypsum was added with stirring. The pregenerated foam mix was next added to the gypsum slurry and stirred to form a foamed gypsum slurry. The slurry was poured into a paper lined mould of 300 x 300 x 12.5 mm dimension and a second sheet of paper placed on top to form a gypsum coupon which was then dried in three stages of successively lower temperatures and longer times to a constant weight. The density and 2 hour water absorption were then determined. The density was calculated by dividing the dry weight of the test specimen by the mould The water absorption was determined by cutting a test specimen measuring 280 x 280 mm from the coupon and immersing this specimen in a water bath at 23°C covered with 25 to 35 mm of water for 2 hours. Its weight before and after immersion was measured and the percentage increase calculated.

The results, which include comparative tests, are shown in figures 1 and 2. In these tests density and 2 hr water absorption were measured and reported using a the emulsifier system of the invention and also as comparison, based on the colloid emulsified system of the above mentioned PCT publication, in which the same amounts of wax emulsion containing an emulsifier system of a bentonite clay and sodium naphthalene sulphonate.

A further example has also been conducted in which the emulsion containing Mobilwax 135, which has a melting point (congealing point) in the range 57-60°C and a maximum content of oil of 1.0wt%, is used at a level of 4% by



weight. This example is then repeated, but using emulsions (at 4% by weight in the gypsum) in which the Mobil wax 135 is replaced by waxes having higher (63-66°C, and 66-69°C, respectively) and lower (54-57°C) melting/congealing points. When used at the same levels in the emulsion, the emulsion (at the same level in the gypsum) produced good results for water absorption. The values were less than 5%, indeed less than 2.5%, in each case.

5

10

15

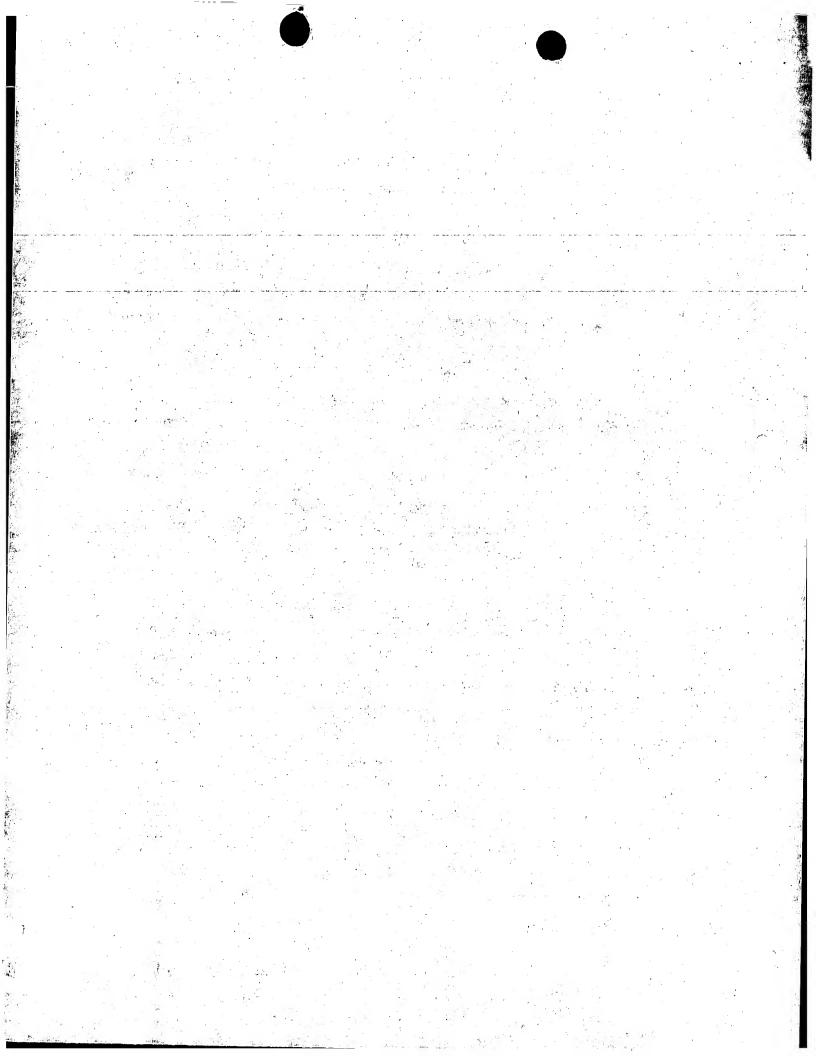
20

25

30

In Figures 3 and 4 there is shown a forming station for gypsum board manufacture is shown generally at 100. It comprises a conveyor 1 which is formed from an array of like, generally coplanar, parallel driven rollers 2 which are rotatable in the same sense. Above the conveyor is a manifold mixing box 3 into which entry conduits 4 and 5 and a plurality of exit nozzles 6,6',6" are flowably connected. A contra-rotatable roller 7 is biased toward rollers 2 to form a nip 8.

In use, a plaster slurry 9 and the pre-generated foam mix 10, detailed above, are supplied under gravity, in an appropriate ratio, though conduits 4 and 5, respectively, into mixing box 3 where they are mixed to form a foamed plaster mixture 11. The foamed plaster mixture is then sprayed through the plurality of nozzles 6,6',6" onto a lower paper liner 12 which is being continuously conveyed, in the direction indicated, by rollers 2. The foamed plaster mixture becomes substantially evenly distributed across, and adheres to, the paper liner. An upper paper liner 13 is continuously conveyed, in the direction indicated by roller 7 into nip 8 where buildup of, the foamed plaster mixture into a dam 9 occurs and the plaster mixture adheres to the paper 13. Uncured plasterboard 14 is continuously conveyed downstream from the forming

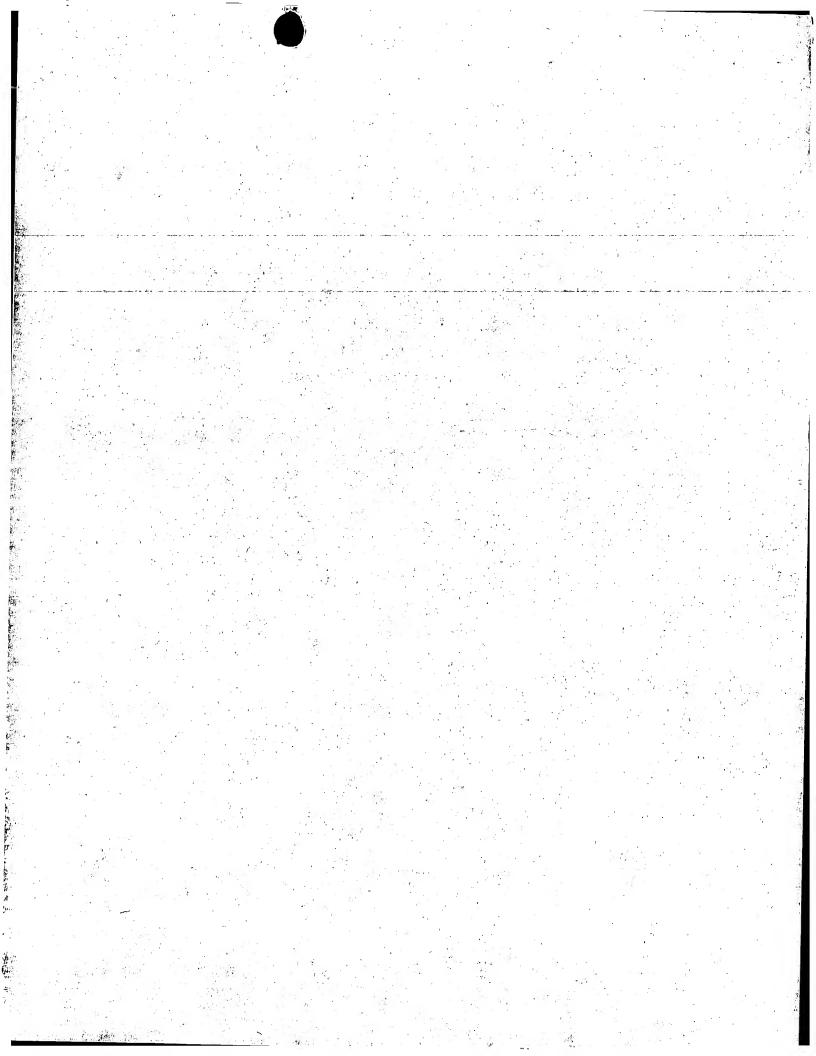


PCT/GB99/00064 WO 99/35103

13

station along a long conveyor belt allowing the chemical reactions of setting to take place. It is then cut to the required length; and dried by passage through multideck drying zones.

In an alternative process, which is not illustrated, a non-foamed slurry is poured into block shaped moulds, in which it is allowed to set partially. The blocks are hard enough to handle after a few minutes, at which time they are pushed from the mould using, for instance, a hydraulic jack, and are conveyed to an oven to complete the 10 setting/drying process.



CLAIMS

5

10

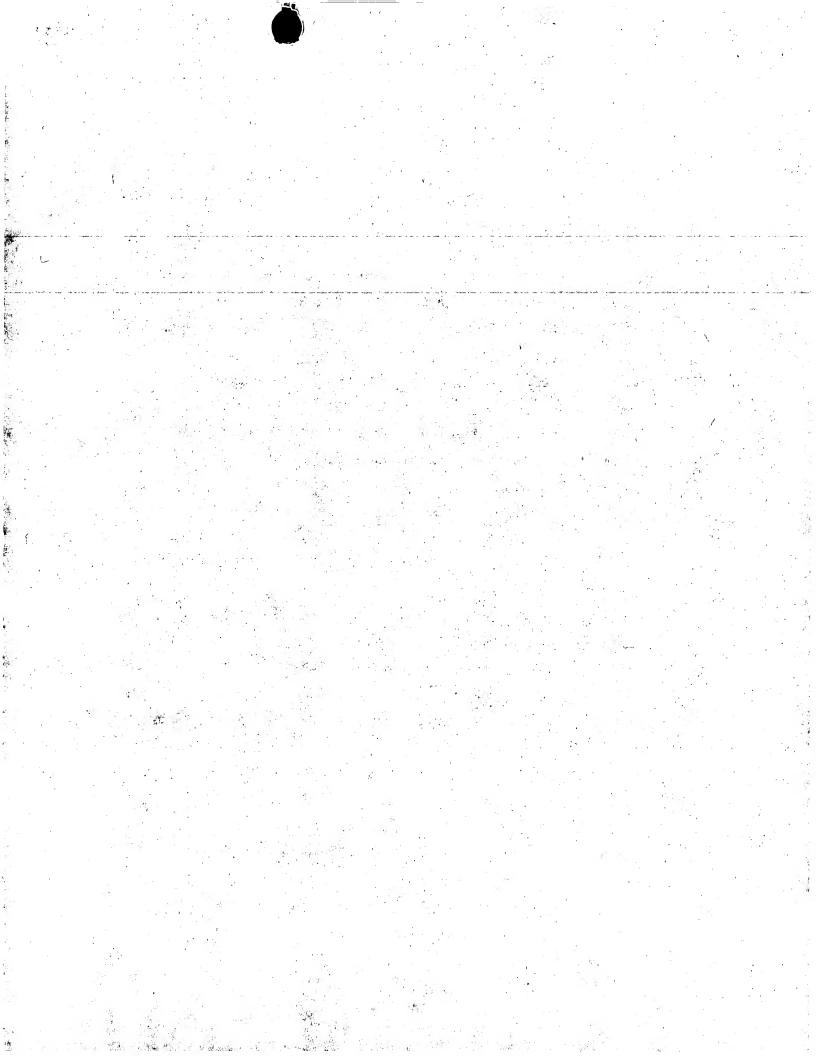
15

20

- 1. A process for producing a gypsum product involving the following steps:
 - a) a slurry of gypsum is formed in water
 - b) the slurry is introduced to mould means and the gypsum allowed to hydrate,

in which a hydrophobing agent comprising an emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system is added to the slurry before introduction into the mould means, and is characterised in that the emulsifier system comprises:

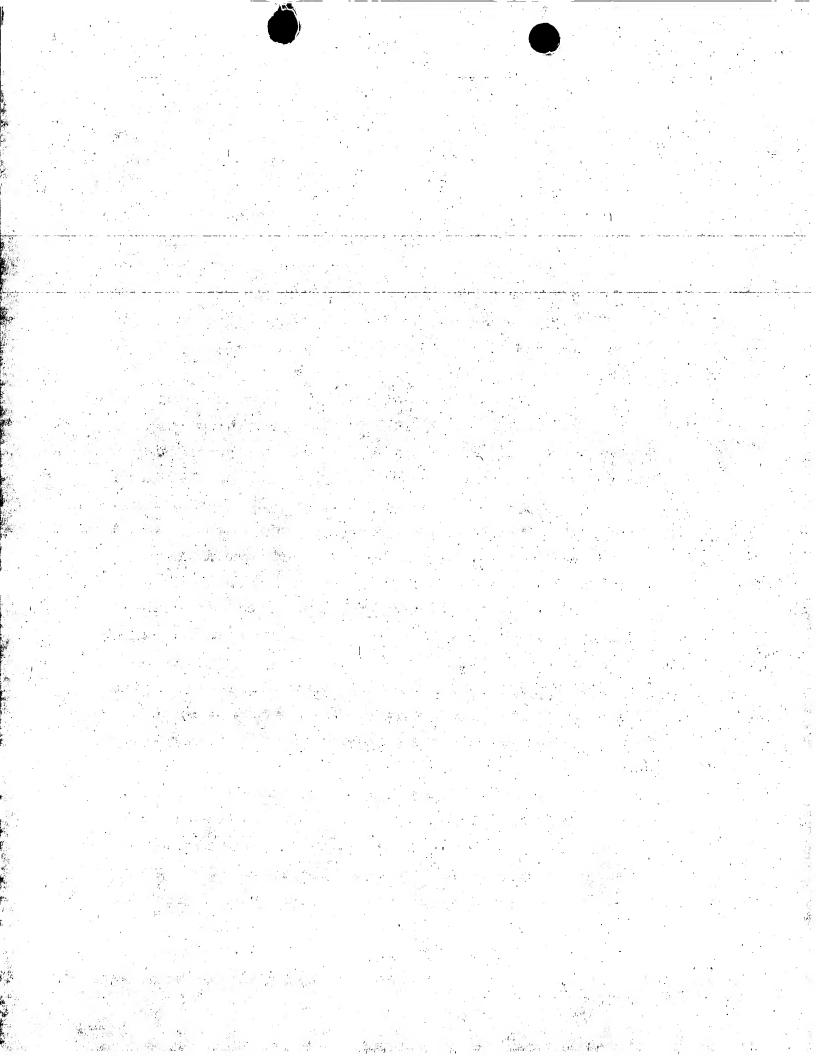
- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
 - ii) an anionic dispersant which is a sulphated compound.
- 2. A process according to claim 1 in which the process is continuous and involves pouring of the slurry onto a continuously moving belt.
- 3. A process according to claim 1 or 2 in which the gypsum slurry is foamed before introduction into the mould means.
- 4. A process according to claim 1 in which the anionic dispersant is a sulphate or a sulphonate.
 - 5. A process according to claim 4 in which the anionic dispersant is a polymeric compound, preferably an aryl sulphonate.
- 6. A process according to claim 5 in which the anionic dispersant is a naphthalene sulphonate, preferably the sodium salt.



10

15

- 7. A process according to any preceding claim in which the nonionic surfactant is a higher alkanol, alkenol, alkanoic or alkenoic acid or aryl alcohol (including phenol) or carboxylic acid ethoxylated with at least 2 equivalents of ethylene oxide, preferably up to 100, for instance 3 to 30 equivalents, ethylene oxide.
- 8. A process according to claim 7 in which the nonionic surfactant is a C_{B-18} -alkanol or -alkenol ethoxylated with 3 to 30 moles of ethylene oxide.
- 9. A process according to any preceding claim in which the petroleum-derived hydrocarbon wax (a) is one with a high melting point and a low oil content, preferably a paraffin wax, more preferably such a wax having a congealing point in the range 55 to 69°C (ASTM D938) a penetration value (by ASTM D1321) at 25°C in the range 10 to 20 mm/10 and at 50°C at least 50 mm/10 and a viscosity at 100°C (by ASTM D445) in the range 3-7 cSt.
 - 10. A process according to any preceding claim in which the montan wax has a congealing point in the range 67-80°C, an acid value (by ASTM D1980) in the range 10 to 37 mgKOH/g, a saponification value (by ASTM D1962) in the range 35 to 100 mgKOH/g, a viscosity (by ASTM D445) at 90°C in the range 20-400 cSt and at 100°C in the range 20 to 200 cSt.
- 25 11. A process according to any preceding claim in which, in the emulsifier, the montan wax is present in an amount in the range 10 to 20% by weight, the hydrocarbon wax is present in an amount in the range 20 to 40% by weight and the emulsifier system is present in an amount in the range 1 to 6% by weight.
 - 12. A process according to any preceding claim in which the ratio of the anionic dispersant to nonionic

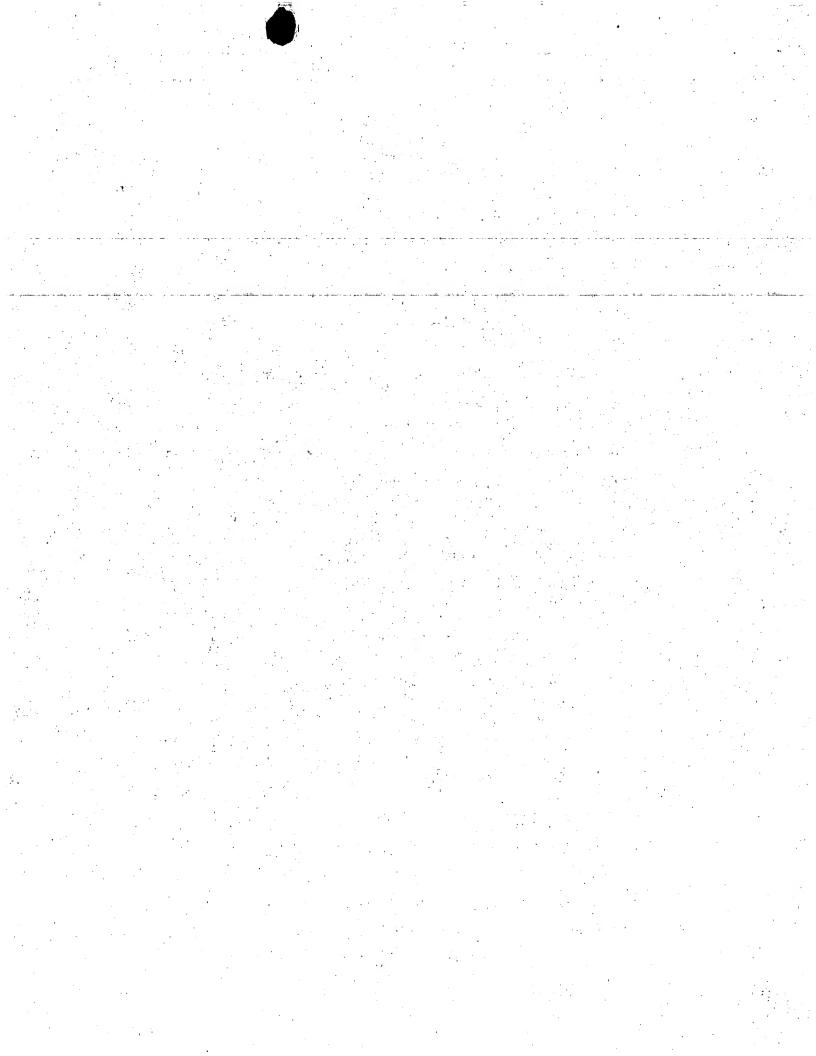


PCT/GB99/00064

20

surfactant in the emulsion is in the range 5:1 to 1:5, preferably 3:1 to 1:3.

- 13. A process according to any preceding claim in which the emulsion is added to the gypsum slurry in an amount in the range 0.5 to 10%, preferably in the range 1.0 to 5.0% by weight based on the weight of gypsum.
- 14. A process according to any preceding claim in which the mould means has a paper liner which becomes permanently laminated to the solidified gypsum.
- 15. A process according to any preceding claim in which the water in which the gypsum slurry is formed has a hardness of at least 100 ppm Ca²⁺, preferably at least 150 ppm Ca²⁺, more preferably at least 200 ppm Ca²⁺.
- 16. An emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system characterised in that the emulsifier system comprises:
 - a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
 - ii) an anionic dispersant which is a sulphated compound.
 - 17. An emulsion according to claim 16 having the further features defined in any of claims 2 to 12.
- 25 18. A method of forming an emulsion in which a petroleum derived hydrocarbon wax and a montan wax are each melted and blended in molten form, an emulsifier system is dissolved into water to form an aqueous emulsifier solution and the molten wax mixture is dispersed into the aqueous emulsifier solution to form an emulsion, characterised in that the emulsifier system comprises:



17

- a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
- ii) an anionic dispersant which is a sulphated compound.
- 19. A method according to claim 18 in which the emulsifier system is as defined in any of claims 2 to 8 and 12 and/or the waxes are as defined in claim 9 and/or claim 10.
- 20. A method according to claim 18 or 19 in which the montan wax is used in an amount in the range 10 to 20% by weight of the emulsion, the hydrocarbon wax is used in an amount in the range 20 to 40% by weight of the emulsion and the emulsifier system is used in an amount in the range 0.5 to 6% by weight, preferably 1 to 2.5% by weight of the emulsion.

20

5



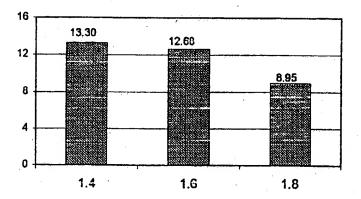


Figure 1

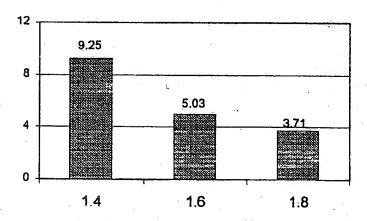


Figure 2



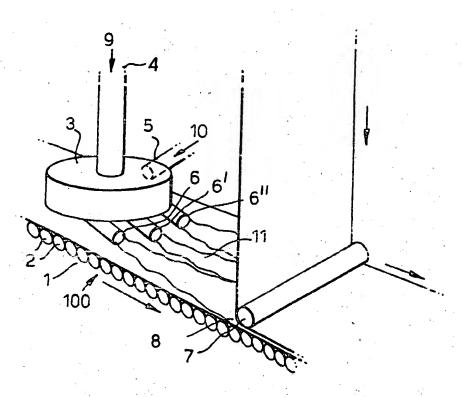


Figure 3

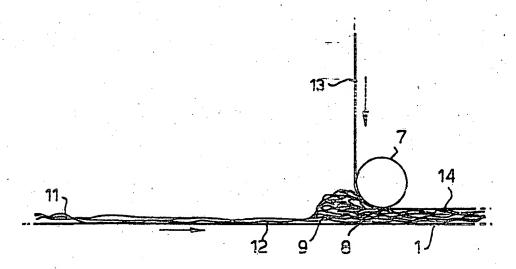
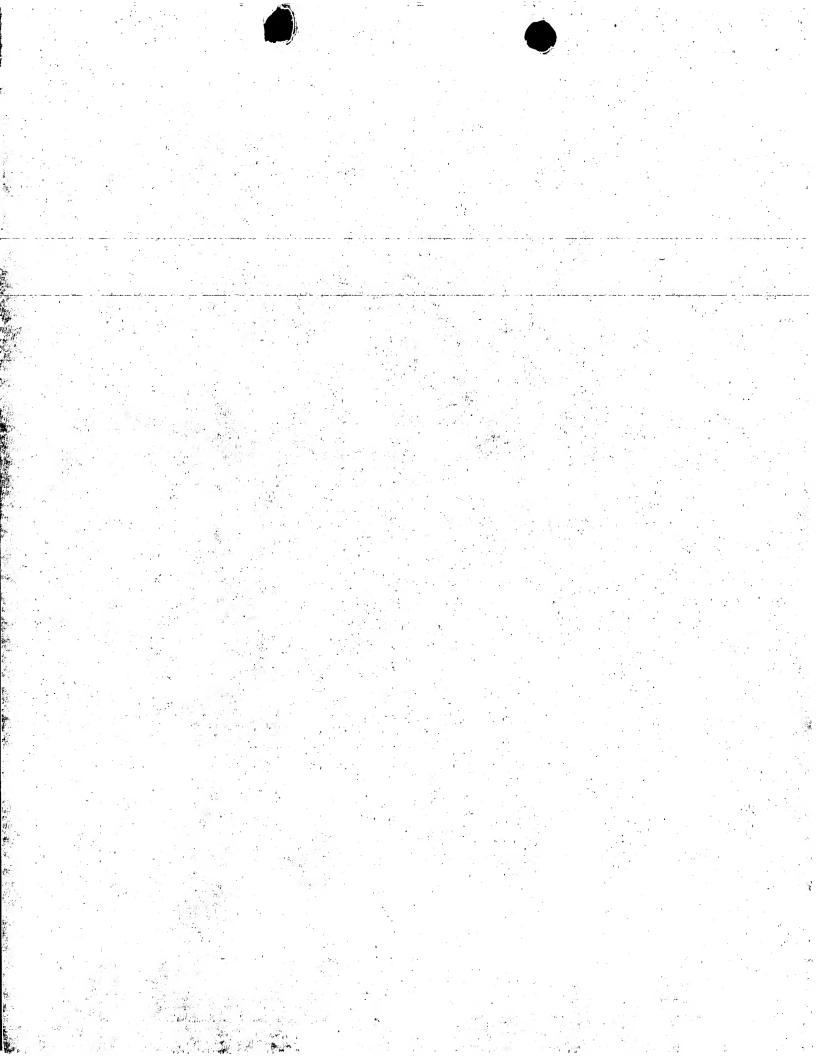


Figure 4





INTERNATIONAL SEARCH REPORT

onal Application No PCT/GB 99/00064

A. CLASSIFICATION OF SUBJECT MATTER IPC 6 C04B28/14 C08L91/06 C04B111:27

//(C04B28/14,24:08,24:22,24:32,24:36),

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

 $\begin{array}{ccc} \text{Minimum documentation searched} & \text{(classification system followed by classification symbols)} \\ IPC & 6 & C04B & C08L \\ \end{array}$

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUM	ENTS CONSIDERED TO BE RELEVANT	
Calegory ^e	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Α ·	US 5 695 553 A (T. CLARET, ET AL.) 9 December 1997 see claim 1	1,4,9, 11,13, 14,16-20
A,P	WO 98 09925 A (MOBIL OIL) 12 March 1998 cited in the application see page 4, line 1-11; claims 1,8	1,3-6
A	US 4 315 957 A (HOECHST AG) 16 February 1982 see column 3, line 6-10 see column 3, line 35-45; claim 1	16,18,20
• • • • • • • • • • • • • • • • • • • •	-/	
	_	
	- -	. •

	
Further documents are listed in the continuation of box C.	X Patent family members are listed in annex.
 Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filling date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosuro, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed 	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive stop when the document is combined with one or more other such documents, such combined with one or more other such documents, such combination being obvious to a person skilled in the art. "&" document member of the same patent family
Date of the actual completion of the international search	Date of mailing of the international search report
22 April 1999	03/05/1999
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentiaan 2 NL - 2280 HV Rijewijk Tel. (+G1-70) 340-2040, Tx. 31 651 epo ni, Fax: (+31-70) 340-3016	Authorized officer Daeleman, P

			· · · · · · · · · · · · · · · · · · ·	*
	100 mm			
			*	4
	*		8)	
			1	
		*		· · ·
	10 to 0	*	, and	
				*)
	* 1 - 2	. 1		
			(1)	
and the second s				
	ed .			0 - 4
		*		, ,
	w.			
*				
	X.			
	ter .		*	
	•			*
			*	
	*			- in the second of the second
		. 1		
	:			
		*	•	
		*		
			•••	v .
	* .			
	* *		* * * * * * * * * * * * * * * * * * * *	
1				
e veneral de la companya de la comp La companya de la co	*			* *
	2			
	X .	* * * * * * * * * * * * * * * * * * * *		* 0
			· •	
			- X -	
				× () =
		0	•	*
	*	and the second s		
		•		•



INTERNATIONAL SEARCH REPORT

Inter onal Application No PCT/GB 99/00064

	Ation) DOCUMENTS CONSIDERED TO BE RELEVANT		7
Category °	Citation of document, with indication, where appropriato, of the relevant passages		Relevant to claim No.
4,P	PATENT ABSTRACTS OF JAPAN vol. 098, no. 008, 30 June 1998 & JP 10 067980 A (MITSUBISHI CHEM CORP), 10 March 1998 see abstract	· · · · · · · · · · · · · · · · · · ·	16
1	US 5 437 722 A (L. BORENSTEIN) 1 August 1995 cited in the application		
1	DATABASE WPI Week 8635 Derwent Publications Ltd., London, GB; AN 86-229176 XP002100852 & JP 61 159494 A (SANWA KAGAKU KK)		*
	see abstract		
			3-4
* \		ν.	
			*
		¥.	
·			

1.

				Mary Mary Mary Mary Mary Mary Mary			4
						3	
							e 1
			1		**		
, ·		*	•				
4				. *			
**	•		Y		•		
						*	
					* * * * * * * * * * * * * * * * * * * *		
		1			*		
							· ·
e.		34			- 44	•	
					*		*
27.0							
A**		1		No.		- O	
200		بالمعطور الساسة المحسا	والمستناف والمستنان المارات المارات	البريب المستريدين المستريد			بدنية هالمان أربسها لمانا
100		•					
8,3		- 0,1					•
100			- 1			* '	
5		ماداليمسي القميس فالمتاب المتوسيين والمستميسية الأم	علامه والمعالم عدد والمساعد	بالعاسفا والمتدوا بالمؤدات فالمطمعيد والمتدار أدا ويسار عاجمة	تا دور فينتفيد منه . دور المدار عنو	العادات السندد والمستدورة المستهدارة المداد	and a management of the same o
G. Y.							
1							* 1
1							*
6							· 1
	* ."			•	*		. 4
		*. 7	7. 4.				** -
ç.					*** (***	*	
direk N	72	*				·	
15 · ·	: 41 ₂			and the second			
17 .					The state of the s		
23					A		3.0
S.			- N				27.00
23							
							*
New Street							
75.							
W.		* *					8
4		* *					
	· B						
	*						8
3							
7	*					*	
· · · · · · · · · · · · · · · · · · ·							
· · · · · · · · · · · · · · · · · · ·							
が 一般の							
· · · · · · · · · · · · · · · · · · ·							
· · · · · · · · · · · · · · · · · · ·							
· · · · · · · · · · · · · · · · · · ·							
· · · · · · · · · · · · · · · · · · ·							
大学 一大学 一大学 一大学 一大学 一大学 一大学 一大学 一大学 一大学 一							
· · · · · · · · · · · · · · · · · · ·							
· · · · · · · · · · · · · · · · · · ·							
の 一般の 一般の 一般の 一般の 一般の 一般の 一般の 一般の 一般の 一般							
《《···································							
《 · · · · · · · · · · · · · · · · · · ·							
のでは、一般の関するというでは、ないでは、これになっているとのでは、これになっているというできない。 これにない かんしょう かんしょう かんしょう かんしょう しょうしょう しょうしょうしょう しょうしょう しょうしょう しょうしょう しょうしょうしょう しょうしょう しょう							
このである。 神の間で、こので、大きなないでは、このなどでは、これには、これには、これには、これには、これには、これには、これには、これに							
このである。 神の間で、このでは、ないでは、このでは、このでは、このでは、このでは、ないでは、ないでは、ないでは、このでは、このでは、このでは、このでは、このでは、このでは、このでは、この							
のでは、「一般の関する」では、「これのでは、「これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、							
このである。 神の下の からから かんかん かんしょう かんしょう こうきょう ないない かんち なななな カルボル・スター・スター・スター・スター・スター・スター・スター・スター・スター・スター							
と、一切である。 神色的な こうさん 内心 こうかん こうかん こうしょう になる こうかい かんこう ないない ないない ないない ないない ないかい かんしょう しょうしょう かんしょう しょうしょう しょうしょうしょう しょうしょう しょう							
と、「一般の一般の一般の一般の一般の一般の一般の一般の一般の一般の一般の一般の一般の一							
のである。 時間のでは、一般のでは、これのでは、これのでは、これのでは、これのでは、一般の理解をは、これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、これのでは、これの							
のである。 (Mission) できた では、これが、これが、これでは、 これでは、 これでは、 ないでは、 ないでは、 ないでは、 ないでは、 ないでは、 ないでは、 ないでは、 ないでは、 ない はい							

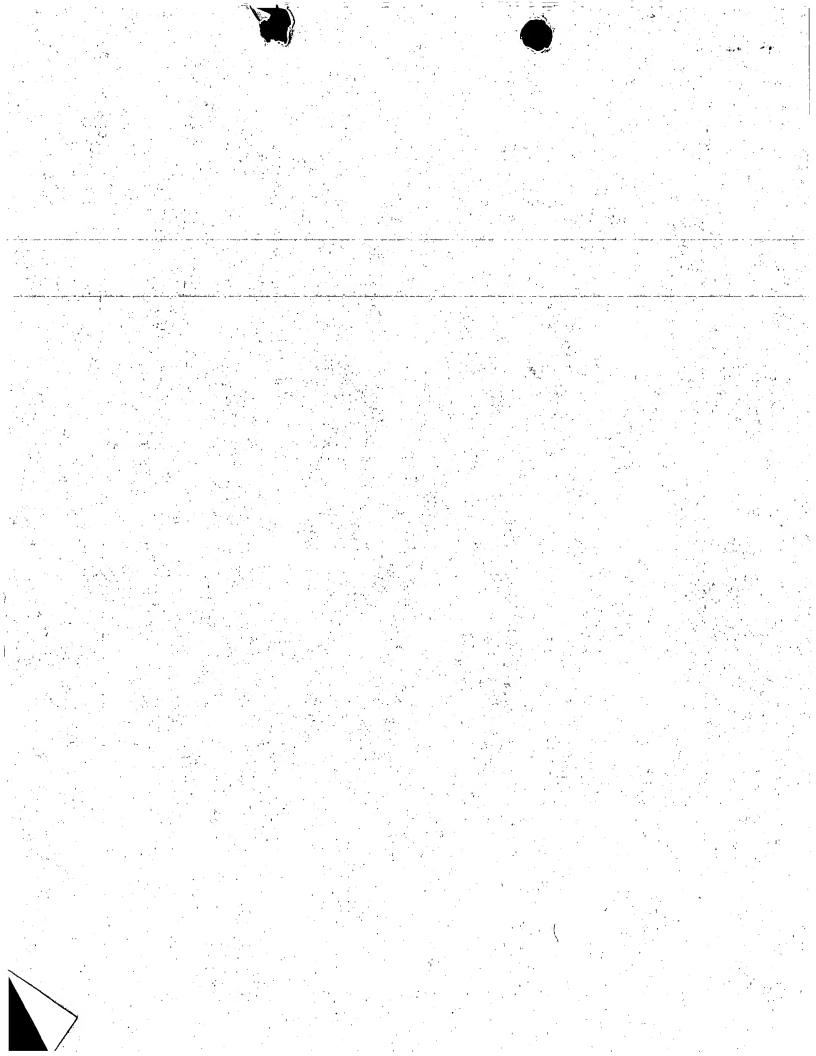


INTERNATIONAL SEARCH REPORT

Information on patent family members

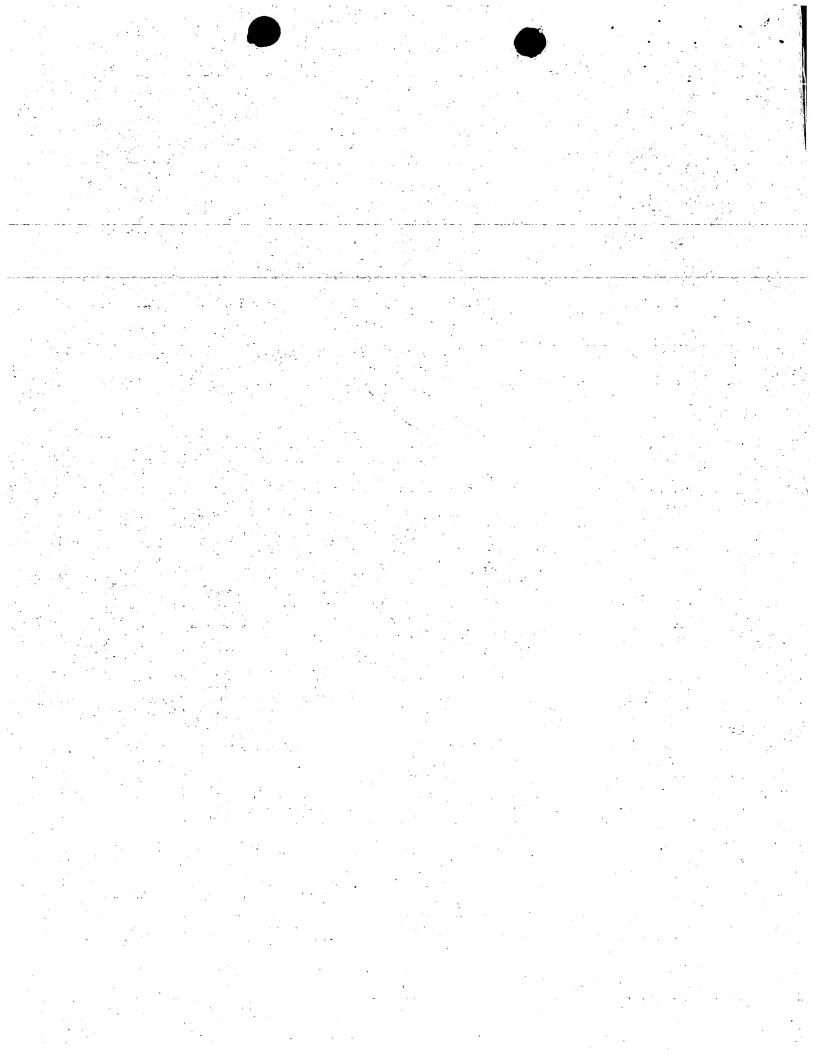
Inter anal Application No PCT/GB 99/00064

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5695553	A 09-12-1997	CA 2184041 A	24-02-1998
WO 9809925	A 12-03-1998	AU 4125397 A	26-03-1998
US 4315957	A 16-02-1982	DE 2926197 A AU 535960 B AU 5972480 A	15-01-1981 12-04-1984 08-01-1981
	,	BR 8004068 A EP 0022212 A JP 56010559 A	21-01-1981 14-01-1981 03-02-1981
* .	vi	JP 63058193 B ZA 8003853 A	15-11-1988 29-07-1981
US 5437722	A 01-08-1995	CA 2116483 A,C EP 0669377 A	30-08-1995
		JP 8337459 A AT 175707 T AU 1637595 A DE 69507174 D	24-12-1996 15-01-1997 24-10-1996 25-02-1999



PTO/PCT Rec'd 07 JUL 2000

From the INTERNATIONAL SEARCHING AUTHORITY	PCT
GILL JENNINGS & EVERY Broadgate House 7 Eldon Street London EC2M 7LH UNITED KINGDOM GILL JENNINGS & EVERY Broadgate House From GILL JENNINGS & EVERY	NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL SEARCH REPORT OR THE DECLARATION (PCT Rule 44.1)
PIAKIEU GILL SERVINGO & A	Date of mailing (day/month/year) 03/05/1999
Applicant's or agent's file reference HMJ03045W0	FOR FURTHER ACTION See paragraphs 1 and 4 below
International application No. PCT/GB 99/00064	International filing date (day/month/year) 08/01/1999
MOBIL OIL COMPANY LIMITED et al.	
1. X The applicant is hereby notified that the International Search Filing of amendments and statement under Article 19: The applicant is entitled, if he so wishes, to amend the claims When? The time limit for filing such amendments is normal International Search Report; however, for more det Where? Directly to the International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Fascimile No.: (41-22) 740.14.35	s of the International Application (see Rule 46):
For more detailed instructions, see the notes on the accordance. The applicant is hereby notified that no International Search Article 17(2)(a) to that effect is transmitted herewith.	
3. With regard to the protest against payment of (an) addition the protest together with the decision thereon has been applicant's request to forward the texts of both the protest.	transmitted to the International Bureau together with the
no decision has been made yet on the protest; the appli	icant will be notified as soon as a decision is made.
4. Further action(s): The applicant is reminded of the following: Shortly after 18 months from the priority date, the international applif the applicant wishes to avoid or postpone publication, a notice priority claim, must reach the International Bureau as provided in completion of the technical preparations for international publication. Within 19 months from the priority date, a demand for international	of withdrawal of the international application, or of the n Rules 90 <i>bis</i> .1 and 90 <i>bis</i> .3, respectively, before the tion.
wishes to postpone the entry into the national phase until 30 mon Within 20 months from the priority date, the applicant must perform before all designated Offices which have not been elected in the priority date or could not be elected because they are not bound	nths from the priority date (in some Offices even later). In the prescribed acts for entry into the national phase I demand or in a later election within 19 months from the
Name and mailing address of the Int rnational Searching Authority European Patent Office, P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Alex Schmidt



PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference HMJ03045W0		of Transmittal of International Search Report 220) as well as, where applicable, item 5 below.
International application No.	International filing date (day/month/year)	(Earliest) Priority Date (day/month/year)
PCT/GB 99/00064	08/01/1999	08/01/1998
Applicant		
MOBIL OIL COMPANY LIMITE	D et al.	
This International Search Report has be according to Article 18. A copy is being	een prepared by this International Searching Aut transmitted to the International Bureau.	hority and is transmitted to the applicant
This International Search Report consis	sts of a total of sheets. by a copy of each prior art document cited in this	s report.
1. Basis of the report		
 With regard to the language, the language in which it was filed, to 	ne international search was carried out on the ba unless otherwise indicated under this item.	sis of the international application in the
the international search Authority (Rule 23.1(b))	was carried out on the basis of a translation of	the international application furnished to this
was carried out on the basis of	and/or amino acid sequence disclosed in the in the sequence listing: tional application in written form.	nternational application, the international search
filed together with the in	nternational application in computer readable for	m.
furnished subsequently	to this Authority in written form.	X
furnished subsequently	to this Authority in computer readble form.	
	subsequently furnished written sequence listing on as filed has been furnished.	loes not go beyond the disclosure in the
the statement that the in furnished	nformation recorded in computer readable form i	s identical to the written sequence listing has been
2. Certain claims were fo	ound unsearchable (See Box I).	· · · · · · · · · · · · · · · · · · ·
3. Unity of Invention is la	acking (see Box II).	
4. With regard to the title ,		•
	submitted by the applicant.	
the text has been estab	lished by this Authority to read as follows:	
		•
	*	
5. With regard to the abstract,		
the text has been estable	submitted by the applicant. lished, according to Rule 38.2(b), by this Authori he date of mailing of this international search rej	ity as it appears in Box III. The applicant may, port, submit comments to this Authority.
	blished with the abstract is Figure No.	
as suggested by the app		None of th figures.
	ailed to suggest a figur .	
	er characterizes the invention.	
Decades this lighte bette	or commentation of the intermedia.	. ·

7		
ja .		
10.		
*		
		1
-4-	t a tampi titama tapia a tapa papa na matakan manda kapangan a tamping ing matakang galaman, anaman a milihana Kanggangkan	
15 8 15		
		المناه كالمرا للواصفات للما
7		·
Pre-		(X)
-		
		- 0
130		
(· · · ·		,
100		
4		
5		
		14
		4
1 m		
કોઇક ંક્ર હ્યું કેંદ્ર		

A. CLASSIFICATION OF SUBJECT MATTER IPC 6 C04B28/14 C08L91/06

CO4B111:27

//(C04B28/14,24:08,24:22,24:32,24:36),

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C04B C08L IPC 6

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Α	US 5 695 553 A (T. CLARET, ET AL.) 9 December 1997	1,4,9, 11,13, 14,16-20
	see claim 1	. * * *
A,P	WO 98 09925 A (MOBIL OIL) 12 March 1998 cited in the application see page 4, line 1-11; claims 1,8	1,3-6
Α	US 4 315 957 A (HOECHST AG) 16 February 1982 see column 3, line 6-10 see column 3, line 35-45; claim 1	16,18,20
• •	-/	- 1
>-		
		•

Patent family members are listed in annex. T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
or priority date and not in conflict with the application but cited to understand the principle or theory underlying the
X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone of the cannot be considered to involve an inventive step when the document is combined with one or more other such document, such combination being obvious to a person skilled in the art.
Date of mailing of the international search report
03/05/1999
Authorized officer Daeleman, P

1





International Application No PCT/GB 99/00064

	on) DOCUMENTS CONSIDERED TO BE RELEVANT Citation of document, with indication, where appropriate, of the relevant passages		Relevant to claim No.	
A,P	PATENT ABSTRACTS OF JAPAN vol. 098, no. 008, 30 June 1998 & JP 10 067980 A (MITSUBISHI CHEM CORP), 10 March 1998 see abstract		16	
\	US 5 437 722 A (L. BORENSTEIN) 1 August 1995 cited in the application			
	DATABASE WPI Week 8635 Derwent Publications Ltd., London, GB; AN 86-229176 XP002100852 & JP 61 159494 A (SANWA KAGAKU KK) see abstract		* .	
				
-		,		
.				
-		•	,	
9 1	*			
,				
			(1)	
		r		
		,		

1

		*				
,			*		,	•
		,	•	· ·		
	***		¥	•		
		(1)				
						s ·
						6
F - 918						
*						
			* * *			
	2			*		
•						
					i de la composición del composición de la compos	
	7		,			
	* . *					
						7 - 7
				one .	79	
	**		**			
						10.0
•		* * * * * * * * * * * * * * * * * * *	**************************************		¥	1 • 12
				in the second se		
	**					
0						
		: W.		· ·	* 4	
			2		* *	
	1				· × *	
		e a				
	***	**************************************			i (with	
		× .		*		**

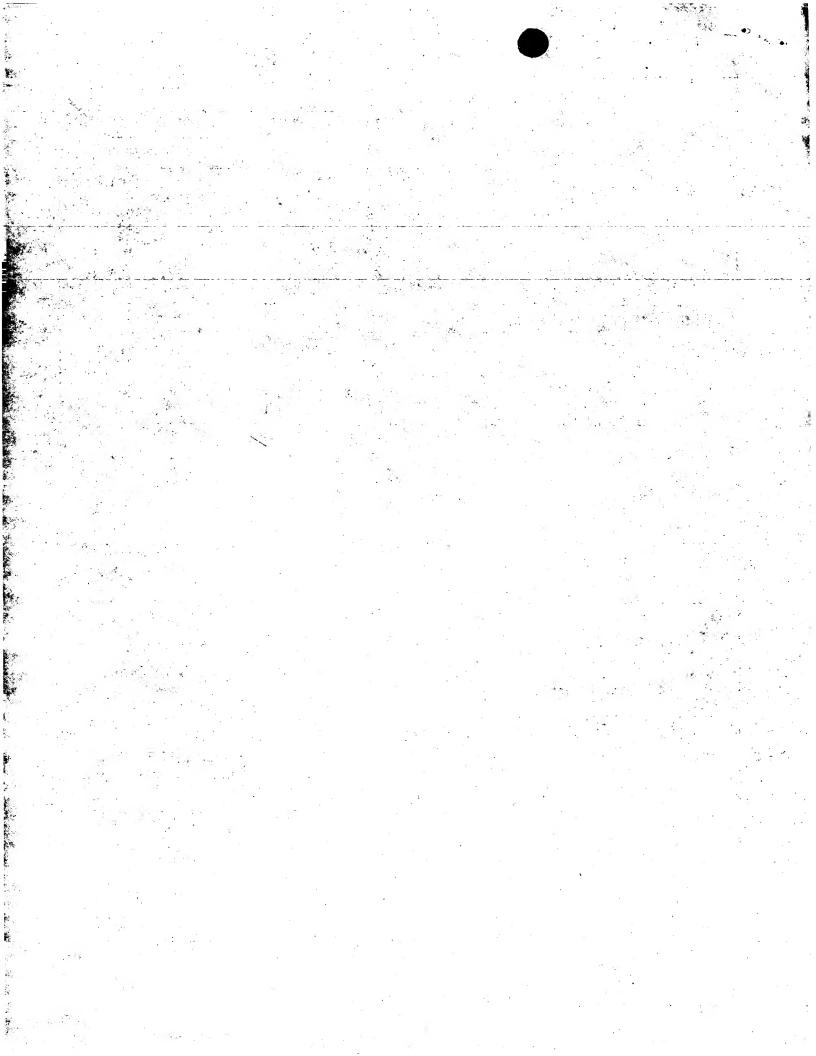
•	* *			, , , , , , , , , , , , , , , , , , ,		, X - 2
		* a (2)			w .	
		*	**			* *
	w.	X	X 1			
	e		• • • · · · · · · · · · · · · · · · · ·			*
			te.			
	- 4	***				

INTERMINIONAL SEARCH REPORT

nformation on patent family members

Internal Application No PCT/GB 99/00064

Patent document cited in search report	Publication . date	Patent family member(s)	Publication date
US 5695553 A	09-12-1997	CA 2184041 A	24-02-1998
WO 9809925 A	12-03-1998	AU 4125397 A	26-03-1998
US 4315957 A	16-02-1982	DE 2926197 A AU 535960 B AU 5972480 A BR 8004068 A EP 0022212 A JP 56010559 A JP 63058193 B ZA 8003853 A	15-01-1981 12-04-1984 08-01-1981 21-01-1981 14-01-1981 03-02-1981 15-11-1988 29-07-1981
US 5437722 A	01-08-1995	AU 676965 B CA 2116483 A,C EP 0669377 A JP 8337459 A AT 175707 T AU 1637595 A DE 69507174 D	27-03-1997 26-08-1995 30-08-1995 24-12-1996 15-01-1997 24-10-1996 25-02-1999



PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6:

C04B 28/14, C08L 91/06 // (C04B 28/14, 24:08, 24:22, 24:32, 24:36), 111:27

(11) International Publication Number:

WO 99/35103

(43) International Publication Date:

15 July 1999 (15.07.99)

(21) International Application Number:

PCT/GB99/00064

(22) International Filing Date:

8 January 1999 (08.01.99)

(30) Priority Data:

9800368.4

8 January 1998 (08.01.98)

GB

(71) Applicant (for all designated States except US): MOBIL OIL COMPANY LIMITED [GB/GB]; Mobil House, 500-600 Witan Gate, Central Milton Keynes, Buckinghamshire MK9 1ES (GB).

(72) Inventor; and

(75) Inventor/Applicant (for US only): BOELEE, Scotia [NZ/GB]; 105 Forest Road, Tunbridge Wells, Kent TN2 5BG (GB).

(74) Agent: GILL JENNINGS & EVERY; Broadgate House, 7 Eldon Street, London EC2M 7LH (GB). (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, Cl, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published

With international search report.

Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: GYPSUM PRODUCT

(57) Abstract

A wax emulsion comprising an emulsifying system containing a sulphated anionic surfactant and a non ionic surfactant having high water solubility (cloud point) and high foaming ability is added to a gypsum slurry to improve the moisture resistance of gypsum board. The wax is a mixture of a petroleum derived hydrocarbon wax and a montan wax.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

Albania	ES	Spain	LS	Lesotho	SI	Slovenia
Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
Austria	FR	France	LU	Luxembourg	SN	Senegal
Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	. TG	Togo
Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
Belgium	. GN	Guinea	MK -	The former Yugoslav	TM	Turkmenistan
Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkey
Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
Benin.	IE ·	Ireland	MN	Mongolia	UA	Ukraine
Brazil	IL	Israel	MR	Mauritania	UG	Uganda
Belarus	IS	Iceland	MW	Malawi	US	United States of America
Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
Central African Republic	JP	Japan	NE	Niger	VN .	Viet Nam
Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
- Switzerland	KG	Kyrgyzstan	NO	Norway	zw	Zimbabwe
Côte d'Ivoire	KP	Democratic People's	NZ	New Zealand		
Cameroon		Republic of Korea	PL	Poland		
China ·	KR	Republic of Korea	PT .	Portugal	2	
Cuba	KZ	Kazakstan	RO	Romania		
Czech Republic	LC	Saint Lucia	RU	Russian Federation		
Germany	LI	Liechtenstein	SD	Sudan	9 .	
Denmark	LK	Sri Lanka	SE	Sweden		
Estonia	LR	Liberia	SG	Singapore		* *
	Armenia Austria Australia Azerbaijan Bosnia and Herzegovina Barbados Belgium Burkina Faso Bulgaria Benin Brazil Belarus Canada Central African Republic Congo Switzerland Côte d'Ivoire Cameroon China Cuba Czech Republic Germany Denmark	Armenia FI Austria FR Australia GA Azerbaijan GB Bosnia and Herzegovina GE Barbados GH Belgium GN Burkina Faso GR Bulgaria HU Benin IE Brazil II Belarus IS Canada IT Central African Republic JP Congo KE Switzerland KG Côte d'Ivoire KP Cameroon China KR Cuba KZ Czech Republic LC Germany LI Denmark LK	Armenia FI Finland Austria FR France Australia GA Gabon Azerbaijan GB United Kingdom Bosnia and Herzegovina GE Georgia Barbados GH Ghana Belgium GN Guinea Burkina Faso GR Greece Bulgaria HU Hungary Benin IE Ireland Brazil IL Israel Belarus IS Iceland Canada IT Italy Central African Republic JP Japan Congo KE Kenya Switzerland KG Kyrgyzstan Côte d'Ivoire KP Democratic People's Cameroon China KR Republic of Korea Cuba KZ Kazakstan Czech Republic LC Saint Lucia Cermany LI Liechtenstein LK Sri Lanka	Armenia FI Finland LT Austria FR France LU Australia GA Gabon LV Azerbaijan GB United Kingdom MC Bosnia and Herzegovina GE Georgia MD Barbados GH Ghana MG Belgium GN Guinea MK Burkina Faso GR Greece Bulgaria HU Hungary ML Benin IE Ireland MN Brazil IL Israel MR Belarus IS Iceland MW Canada IT Italy MX Central African Republic JP Japan NE Congo KE Kenya NL Switzerland KG Kyrgyzstan NO Côte d'Ivoire KP Democratic People's NZ Cameroon REPUBLIC OF KR Cameroon REPUBLIC OF KOREA PL China KR Republic of Korea PT Cuba KZ Kazakstan RO Czech Republic LC Saint Lucia RU Cermany LI Liechtenstein SD Denmark LK Sri Lanka SE	Armenia FI Finland LT Lithuania Austria FR France LU Luxembourg Australia GA Gabon LV Latvia Azerbaijan GB United Kingdom MC Monaco Bosnia and Herzegovina GE Georgia MD Republic of Moldova Barbados GH Ghana MG Madagascar Belgium GN Guinea MK The former Yugoslav Burkina Faso GR Greece Republic of Macedonia Bulgaria HU Hungary ML Mali Benin IE Ireland MN Mongolia Brazil IL Israel MR Mauritania Belarus IS Iceland MW Malawi Canada IT Italy MX Mexico Central African Republic JP Japan NE Niger Congo KE Kenya NL Netherlands Switzerland KG Kyrgyzstan NO Norway Côte d'Ivoire KP Democratic People's NZ New Zealand Cameroon Republic of Korea PL Poland China KR Republic of Korea PL Portugal Cuba KZ Kazakstan RO Romania Czech Republic LC Saint Lucia RU Russian Federation Germany LI Liechtenstein SD Sudan Denmark LK Sri Lanka SE Sweden	Armenia FI Finland LT Lithuania SK Austria FR France LU Luxembourg SN Australia GA Gabon LV Latvia SZ Azerbaijan GB United Kingdom MC Monaco TD Bosnia and Herzegovina GE Georgia MD Republic of Moldova TG Barbados GH Ghana MG Madagascar Tj Belgium GN Guinea MK The former Yugoslav TM Burkina Faso GR Greece Republic of Macedonia TR Bulgaria HU Hungary ML Mali TT Benin IE Ireland MN Mongolia UA Brazil IL Israel MR Mauritania UG Belarus IS Iceland MW Malawi US Canada IT Italy MX Mexico UZ Central African Republic JP Japan NE Niger VN Congo KE Kenya NL Netherlands YU Switzerland KG Kyrgyzstan NO Norway ZW Côte d'Ivoire KP Democratic People's NZ New Zealand Cameroon Republic of Korea PL Potand China KR Republic of Korea PT Portugal Cuba KZ Kazakstan RO Romania Czech Republic LC Saint Lucia RU Russian Federation Germany LI Liechtenstein SD Sudan Denmark LK Sri Lanka SE Sweden

WO 99/35103

10

15

20

25

2/PRTS

PCT/GB99/00064

GYPSUM PRODUCT

This invention relates to a gypsum product and to a process for its manufacture. More particularly, this invention relates to a foamed gypsum product of improved water resistance and/or reduced density and to a process, preferably to a continuous process, for its manufacture.

Gypsum board (or plaster board or wallboard) is used extensively in the construction industry. It typically comprises a substantially flat core of set gypsum on either side of which a liner may be adhered. A liner typically comprises paper. The core may be reinforced; for example, reinforced with glass fibres.

Gypsum products (or Plaster of Paris or plaster products) are produced by mixing anhydrous calcium sulphate or calcium sulphate hemihydrate with water, and permitting the mixture to set thereby producing calcium sulphate dihydrate. Often the slurry is foamed by incorporating a preformed solution of foaming agent in water (a surface active material) before adding to the mould means. pervasive problem with gypsum products, however, is that calcium sulphate dihydrate absorbs water and this reduces the strength of the gypsum product. Because of this, plaster board (for example) is required, at least in uses where a relatively high humidity is anticipated (for example, kitchens or bathrooms) to be substantially moisture resistant and this requires the presence of a hydrophobing agent. ("Hydrophobing" is a term used in the art to denote a method of preventing, or reducing water absorption).

30 Silicone oil has previously been used as a hydrophobing agent for gypsum products. It is, however, expensive and in relatively short supply. It also has

20

30

834 Rec Protest Continues

. .

difficulty in providing a moisture resistance of less than 5 wt % water absorption in the test hereinafter mentioned.

In US-A-5437722 an aqueous emulsion comprising a hydrocarbon wax, a montan wax and emulsifier/stabiliser system and also including a polyvinyl alcohol, is used to render gypsum products water resistant. The emulsifier system may include non-ionic or anionic surfactant and alkali. Examples of non ionic surfactants are alkylphenoxypoly(ethyleneoxy) ethanols, sorbitan fatty acid esters and polyoxyethylene sorbitan fatty acid esters. Examples of anionic surfactants are saponified fatty acids.

In our copending application number PCT/GB97/02366, unpublished at the priority date of the present invention, we describe a foamed gypsum product which is hydrophobed by incorporation of an aqueous emulsion comprising a hydrocarbon wax, a montan wax and a colloid stabilised emulsifier system. The preferred colloid stabilised emulsifier system comprises either organic or, more preferably, inorganic colloidal materials. One example is a montmorillonite clay based system in combination with a sodium naphthalene sulphonate.

In the above mentioned PCT application, a comparative example uses an emulsifier system based on a combination of a nonionic surfactant, which was not specifically defined, with an anionic surfactant again, not specifically defined. The emulsifier system used in that comparative example was in fact a combination of an alkyl phenyl ethoxylate with a soap-type anionic surfactant. The worked example using that emulsifier system did not work. In fact it is now believed that the anionic surfactant caused collapse of the foam or that, upon addition to a slurry formed using

15

20

25

30

relatively hard water, the anionic surfactant was precipitated out of the system by the hardness ions.

The above mentioned PCT application also discloses a comparative example using a nonionic surfactant based emulsifier system including no anionic emulsifier. Whilst this gave some improvement over the mixed system, it was found that using a high enough level of emulsion in the gypsum product to achieve adequate density and/or water adsorption figures lead to over-wetting of paper used for the gypsum product and delamination during production.

In the present invention there is a provided a process for producing a foamed gypsum product involving the following steps:

- a) a slurry of gypsum is formed in water
- b) the slurry is introduced to mould means and allowed to hydrate,

in which a hydrophobing agent comprising an emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system is added to the slurry before introduction into the mould means, and is characterised in that the emulsifier system comprises:

- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point (in saline per DIN 53917) of at least 50; and
- ii) an anionic dispersing agent which is a sulphated compound.

Preferably the anionic dispersant is a so called acid stable compound, that is the compound is ionised over a wide range of pH's including acidic pH. The acid stability can be judged by the pK_a of the conjugate acid, which should preferably be less than 4, more preferably less than

4

3, for instance 2 or less. The anionic dispersant is generally a sulphate or a sulphonate.

A suitable class of anionic dispersants are sulphated naphthalene/formaldehyde condensates, for instance having molecular weight in the range 6000 to 40000. These compounds are also known as naphthalene sulphonates. Other aryl sulphonates may also be used. The anionic dispersant is generally used in the emulsion in the form of its sodium salt. Alternatively potassium, ammonium, or even divalent metal salts such as calcium or magnesium, may be used. Suitable compounds are available from BASF AG under the trade name Tamol (trademark).

5

10

15

20

25

30

The nonionic surfactant must be relatively water soluble. The water solubility of non ionic surfactants can be determined by standard test method DIN 53917 in saline. The component should have a cloud point of at least 50, for instance more than 60, up to around 100, for instance approximately 75.

We have found that the nonionic surfactant giving optimum performance is one which has a high foaming ability. Foaming ability can be measured by standard test methods DIN 53902. For instance the test should be carried out according to the method given in sheet 1 of that standard test method, at 40°C, with the surfactant being used in a concentration of 2 g/l in water containing 1.8 mmol Ca ions/l, the duration of the test being 30 seconds. The foaming ability should be at least 300, more preferably at least 500, for instance up to 750. Nonionic surfactants with foaming ability using the above mentioned test method of around 600 are available.

The nonionic surfactant is generally an ethoxylated higher alkyl, alkenyl, alkanoyl or alkenoyl compound.

5

Alternatively ethoxylated aryl compounds may be used, for instance ethoxylated alkyl phenol derivatives. Preferably the compound is a C_{8-18} -alkanol ethoxylated with 3 to 30 equivalents of ethylene oxide, for instance a C_{12-18} -alkanol ethoxylated with 10 to 20 equivalents of ethylene oxide. Suitable compounds are available under the trade name Lutensol (trademark) from BASF AG. It may alternatively be possible to use polyglycosylated alkyl, alkenyl, alkanoyl, alkenoyl and aryl compounds for instance alkyl polyglucosides.

10

15

20

25

30

The use of an acid stable anionic dispersant, it is believed, avoids the addition of the emulsion resulting in the collapse of the foam in the gypsum slurry. Accordingly the density of the product is optimised. The use of the anionic dispersant in combination with non ionic surfactant avoids the use of such high concentrations of nonionic dispersant in the gypsum slurry which can lead to overwetting of paper used in the gypsum product delamination during production. The preferred surfactant makes the emulsion suitable for use with gypsum slurries made up in hard water, for instance water having hardness value of at least 100 ppm Ca2+ even more than 150 ppm Ca2+1, for instance at least 200 ppm Ca2+.

The use of the high foaming non ionic surfactant is believed also to contribute to optimisation of the gypsum slurry foam and the density of the final product. The preferred combination of surfactants in the emulsifier system allows high levels of wax to be incorporated into the final product for optimum hydrophobing of the gypsum product.

The emulsifier system has process advantages also in production of non-foamed products, for instance, other

products made on continuous lines such as fire resistance board. The system provides good compatibility with the equipment

The petroleum-derived hydrocarbon wax (a) is preferably one with a high melting point and a low oil content. A preferred such wax is a paraffin wax, such as fully refined paraffin wax. Fully refined paraffin waxes are generally obtained from highly paraffinic refinery streams such as those obtained from the solvent dewaxing of distillates and other lube fractions. The product is further typically characterised as follows:

CHARACTERISTICS	TEST METHOD	SPECIFICATION		
		MIN	MAX	
Congealing Point (°C)	ASTM D938	55	69	
Oil in Wax (%)	ASTM D721		1	
Penetration at 25°C (mm/10)	ASTM D1321	10	20	
Penetration at 50°C (mm/10)	ASTM D1321		80	
Viscosity (cSt @ 100°C)	ASTM D445	3	7	

25

30

20

15

An example of a fully refined paraffin wax which has been found to be entirely satisfactory, and which satisfies the above specification, is MOBILWAX 135 (derived from the 150 SPN stream) as supplied by Mobil Oil Company Limited; MOBILWAX 145 or 150 (derived from the 300 or 450 SPN stream) are also suitable. While these waxes are hydrofinished to give a white colour and good odour, unfinished wax (which differs only in colour and odour) is also suitable for use in accordance with the invention.

The petroleum-derived hydrocarbon wax a) suitably comprises from 20 to 40 wt % of the aqueous emulsion, preferably from 25 to 35 wt % of the aqueous emulsion.

The montan wax or lignite wax b) is another wax with a high melting point. It is preferably used in crude (or raw) form. Such a product is typically characterised as follows:

CHARACTERISTICS	TEST METHOD	SPECIFICATION		PREFERRE SPECIFIC ATION	
		MIN	MAX	MIN	м
Congealing Point (°C)	ASTM D938	67	80	75	1
Acid Value (mg KOH/g)	ASTM D1980	10	37	10	;
Saponification Value (mgKOH/g)	ASTM D1962	35	100	65	
Ash Content (% wt)	ASTM D482		1		1
Density at 20°C (g/cm³)	ASTM D1298	0.95	1.04	0.95	.1 .
Viscosity (cSt at 90°C)	ASTM D445	20	400	150	4
Viscosity (cSt at 100°C)	ASTM D445	20	200	60	1

25

30

The montan wax b) suitably comprises from 10 to 20 wt % of the aqueous emulsion, preferably from 11 to 15 wt % of the aqueous emulsion.

The emulsifier system (i.e. the total of two or more components of a multi-component system) is suitably present in an amount from 0.5 to 6 wt %, preferably 12 to 5 wt %, more preferably 1.5 to 4% of the aqueous emulsion. The

8

ratio of the non-ionic and anionic components is preferably in the range 5:1 to 1:5, more preferably 3:1 to 1:3, most preferably 2:1 to 1:2.

In the invention the slurry in water, preferably contains 100 parts by weight of gypsum and from 0.5 to 10, preferably from 1 to 5 % by weight of an emulsion as herein defined. The slurry suitably contains 50-60 weight % gypsum and 40-50 weight % water, preferably about 55% gypsum. An accelerator is usually added, for instance a slurry mix from a previous batch.

5

20

30

The slurry preferably contains a foaming agent. Preferably the product is a paper lined board and the process thus preferably includes a step of foaming, usually involving formation of a pre-formed foam by vigorous stirring of the foaming agent in water, followed by mixing the prefoam into preformed gypsum slurry containing the emulsion.

This invention also provides a water-resistant gypsum product which comprises the set composition. Whilst the product may be an unlined board, the invention is particularly applicable to a product which comprises a core product of a set such composition sandwiched between a pair of liners usually paper liners. Another suitable product is fire resistant board which has a glass fibre scrim embedded in each surface of the gypsum board, which is generally unfoamed. The invention includes also the emulsion itself and the process for making it.

The invention further provides a process for the preparation of a water-resistant gypsum board product, which process comprises forming a mixture which is a slurry in water containing 100 parts by weight of gypsum and from 0.5 to 5 parts by weight of an emulsion according to the

5

10

15

20

25

30

9

invention; forming a layer of the mixture in a mould means and drying the layer of gypsum mixture, while permitting hydration of the gypsum, for form a board product. Preferably the process is continuous. The process may be for forming tiles or blocks or boards. Blocks may be formed in moulds from which they are removed when set. Tiles or boards may be formed by spreading a layer of the gypsum mixture on a first planar substrate, a second planar substrate is positioned over the layer to form an assembly, and the mixture is allowed to set in the assembly. gasket may be provided between the planar substrates. Where the product is a lined board, the first and second planar substrates are constituted by liner, for instance paper, usually supported in a mould. Where the product is to be unlined, the planar substrates are removed when the product is set. Where the product is a block, it is usually removed from a mould before the mixture is completely set, but when it is hard enough to handle. Where the product is a fire resistant board a fibreglass scrim is embedded in each side of the slurry in the mould means by feeding to each side of the poured (unfoamed) slurry before the nip forming a dam in an apparatus similar to that shown in Figures 3 and 4 below.

This invention includes the use of an aqueous emulsion of the invention to furnish a gypsum product with water resistance and the use of an aqueous emulsion of the invention to aid foaming of a gypsum slurry, for instance to reduce the density of the set foamed gypsum product.

Figure 1 shows the water absorption results of the product of the comparative example.

Figure 2 shows the water absorption results of the product of the example of the invention.

10

Figures 3 and 4 are a schematic representations of a foaming station for lined gypsum board.

The following Example illustrate the invention.

EXAMPLE

5.

10

25

First the wax phase was prepared by dissolving 12 wt % crude montan wax (Crude Montan Wax supplied by Schuemann Sabol GmbH) in 30 wt % of fully refined paraffin wax (MOBILWAX 135 supplied by Mobil Oil Company Limited) at a raised temperature. 1% by weight sodium naphthalene sulphonate (a sulphated naphthalene formaldehyde condensate having a molecular weight of about 6000 to 40000) from the TAMOL (trademark) range supplied by BASF was added to water to form the aqueous phase and stirred for a period. 0.45% Non ionic surfactant (added as a 90% aqueous solution) (a C13 alkanol - 12 mole ethoxylate available as Lutensol TO12 series) was then added to the aqueous phase and stirred for a period. The temperature of the wax melt was lowered to 100°C and the wax phase was next added to the aqueous phase heated to a suitable temperature, with stirring for a suitable period to form a pre-emulsion. The pre-emulsion still at a temperature was next recycled through a homogeniser, with no impressed pressure, for a full pass. Gradually, the pressure was increased to a value in the range 20-25 MPa (220 bar) and the emulsion recycled for a further pass to form an aqueous emulsion in accordance with the invention.

The emulsion was then tested for its performance in the production of a gypsum product. A conventional foaming agent was mixed with vigorous stirring with a suitable quantity water to generate a foam mixture. A gypsum slurry mix was prepared by adding a predetermined amount (1.4, 1.6 or 1.8% by weight based on the amount of gypsum) of wax

10

15

20

25

30

emulsion (according to the invention or comparative) to around 40 parts by weight water along with predetermined amounts of a wetting agent, starch and an accelerator in a total amount of 0.38 parts by weight. To this around 58 parts by weight gypsum was added with stirring. The pregenerated foam mix was next added to the gypsum slurry and stirred to form a foamed gypsum slurry. The slurry was poured into a paper lined mould of 300 x 300 x 12.5 mm dimension and a second sheet of paper placed on top to form a gypsum coupon which was then dried in three stages of successively lower temperatures and longer times to a constant weight. The density and 2 hour water absorption were then determined. The density was calculated by dividing the dry weight of the test specimen by the mould The water absorption was determined by cutting a test specimen measuring 280 x 280 mm from the coupon and immersing this specimen in a water bath at 23°C covered with 25 to 35 mm of water for 2 hours. Its weight befor and after immersion was measured and the percentage increase calculated.

The results, which include comparative tests, are shown in figures 1 and 2. In these tests density and 2 hr water absorption were measured and reported using a the emulsifier system of the invention and also as comparison, based on the colloid emulsified system of the above mentioned PCT publication, in which the same amounts of wax emulsion containing an emulsifier system of a bentonite clay and sodium naphthalene sulphonate.

A further example has also been conducted in which the emulsion containing Mobilwax 135, which has a melting point (congealing point) in the range 57-60°C and a maximum content of oil of 1.0wt%, is used at a level of 4% by

30

weight. This example is then repeated, but using emulsions (at 4% by weight in the gypsum) in which the Mobil wax 135 is replaced by waxes having higher (63-66°C, and 66-69°C, respectively) and lower (54-57°C) melting/congealing points. When used at the same levels in the emulsion, the emulsion (at the same level in the gypsum) produced good results for water absorption. The values were less than 5%, indeed less than 2.5%, in each case.

In Figures 3 and 4 there is shown a forming station for gypsum board manufacture is shown generally at 100. It comprises a conveyor 1 which is formed from an array of like, generally coplanar, parallel driven rollers 2 which are rotatable in the same sense. Above the conveyor is a manifold mixing box 3 into which entry conduits 4 and 5 and a plurality of exit nozzles 6,6',6" are flowably connected. A contra-rotatable roller 7 is biased toward rollers 2 to form a nip 8.

In use, a plaster slurry 9 and the pre-generated foam mix 10, detailed above, are supplied under gravity, in an appropriate ratio, though conduits 4 and 5, respectively, into mixing box 3 where they are mixed to form a foamed The foamed plaster mixture is then plaster mixture 11. sprayed through the plurality of nozzles 6,6',6" onto a lower paper liner 12 which is being continuously conveyed, in the direction indicated, by rollers 2. The foamed plaster mixture becomes substantially evenly distributed across, and adheres to, the paper liner. An upper paper liner 13 is continuously conveyed, in the direction indicated by roller 7 into nip 8 where buildup of, the foamed plaster mixture into a dam 9 occurs and the plaster mixture adheres to the paper 13. Uncured plasterboard 14 is continuously conveyed downstream from the forming

10

station along a long conveyor belt allowing the chemical reactions of setting to take place. It is then cut to the required length; and dried by passage through multideck drying zones.

In an alternative process, which is not illustrated, a non-foamed slurry is poured into block shaped moulds, in which it is allowed to set partially. The blocks are hard enough to handle after a few minutes, at which time they are pushed from the mould using, for instance, a hydraulic jack, and are conveyed to an oven to complete the setting/drying process.

CLAIMS

10.

15

20

- 1. A process for producing a gypsum product involving the following steps:
 - a) a slurry of gypsum is formed in water
 - b) the slurry is introduced to mould means and the gypsum allowed to hydrate,

in which a hydrophobing agent comprising an emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system is added to the slurry before introduction into the mould means, and is characterised in that the emulsifier system comprises:

- a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
- ii) an anionic dispersant which is a sulphated compound.
- 2. A process according to claim 1 in which the process is continuous and involves pouring of the slurry onto a continuously moving belt.
- 3. A process according to claim 1 or 2 in which the gypsum slurry is foamed before introduction into the mould means.
- 4. A process according to claim 1 in which the 25 anionic dispersant is a sulphate or a sulphonate.
 - 5. A process according to claim 4 in which the anionic dispersant is a polymeric compound, preferably an aryl sulphonate.
 - 6. A process according to claim 5 in which the anionic dispersant is a naphthalene sulphonate, preferably the sodium salt.

PCT/GB99/00064

5

10

15

20

- 7. A process according to any preceding claim in which the nonionic surfactant is a higher alkanol, alkenol, alkanoic or alkenoic acid or aryl alcohol (including phenol) or carboxylic acid ethoxylated with at least 2 equivalents of ethylene oxide, preferably up to 100, for instance 3 to 30 equivalents, ethylene oxide.
- 8. A process according to claim 7 in which the nonionic surfactant is a C_{8-18} -alkanol or -alkenol ethoxylated with 3 to 30 moles of ethylene oxide.
- 9. A process according to any preceding claim in which the petroleum-derived hydrocarbon wax (a) is one with a high melting point and a low oil content, preferably a paraffin wax, more preferably such a wax having a congealing point in the range 55 to 69°C (ASTM D938) a penetration value (by ASTM D1321) at 25°C in the range 10 to 20 mm/10 and at 50°C at least 50 mm/10 and a viscosity at 100°C (by ASTM D445) in the range 3-7 cst.
- 10. A process according to any preceding claim in which the montan wax has a congealing point in the range 67-80°C, an acid value (by ASTM D1980) in the range 10 to 37 mgKOH/g, a saponification value (by ASTM D1962) in the range 35 to 100 mgKOH/g, a viscosity (by ASTM D445) at 90°C in the range 20-400 cSt and at 100°C in the range 20 to 200 cSt.
- 25 11. A process according to any preceding claim in which, in the emulsifier, the montan wax is present in an amount in the range 10 to 20% by weight, the hydrocarbon wax is present in an amount in the range 20 to 40% by weight and the emulsifier system is present in an amount in the range 1 to 6% by weight.
 - 12. A process according to any preceding claim in which the ratio of the anionic dispersant to nonionic

surfactant in the emulsion is in the range 5:1 to 1:5, preferably 3:1 to 1:3.

- 13. A process according to any preceding claim in which the emulsion is added to the gypsum slurry in an amount in the range 0.5 to 10%, preferably in the range 1.0 to 5.0% by weight based on the weight of gypsum.
 - 14. A process according to any preceding claim in which the mould means has a paper liner which becomes permanently laminated to the solidified gypsum.
- 15. A process according to any preceding claim in which the water in which the gypsum slurry is formed has a hardness of at least 100 ppm Ca²⁺, preferably at least 150 ppm Ca²⁺, more preferably at least 200 ppm Ca²⁺.
 - 16. An emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system characterised in that the emulsifier system comprises:
 - a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
 - ii) an anionic dispersant which is a sulphated compound.
 - 17. An emulsion according to claim 16 having the further features defined in any of claims 2 to 12.

20

18. A method of forming an emulsion in which a petroleum derived hydrocarbon wax and a montan wax are each melted and blended in molten form, an emulsifier system is dissolved into water to form an aqueous emulsifier solution and the molten wax mixture is dispersed into th aqueous emulsifier solution to form an emulsion, characterised in that the emulsifier system comprises:

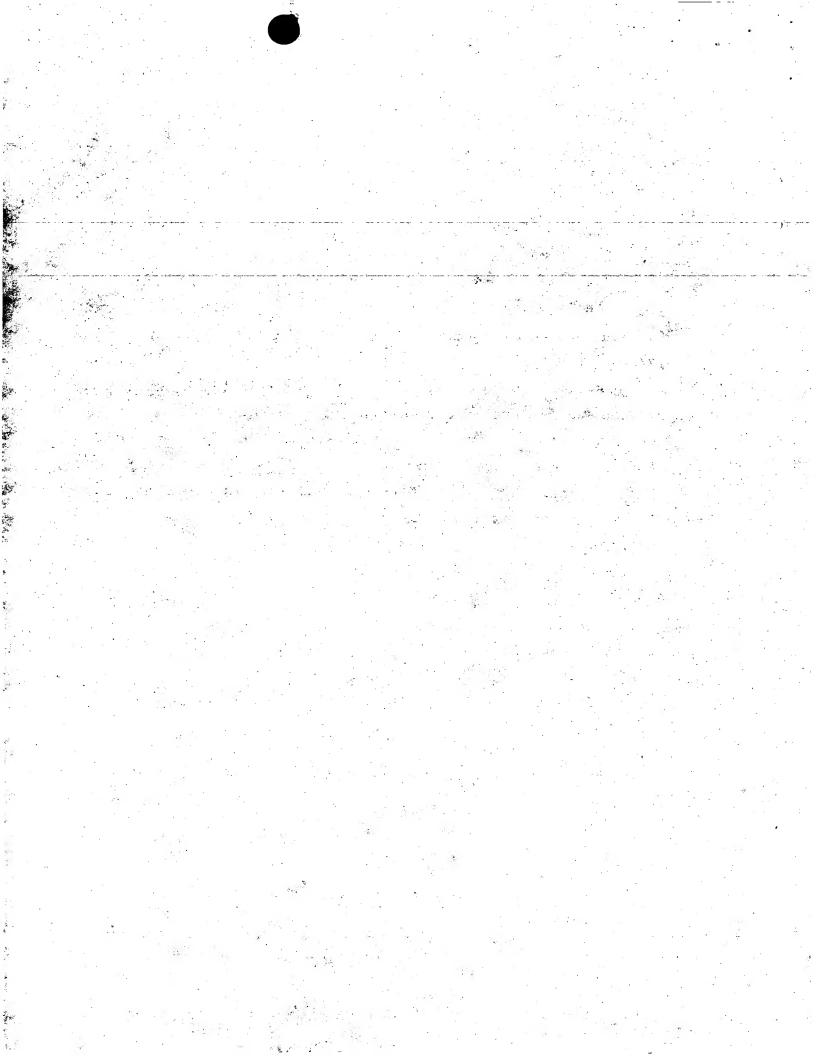
PCT/GB99/00064

5

- a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
- ii) an anionic dispersant which is a sulphated compound.
- 19. A method according to claim 18 in which the emulsifier system is as defined in any of claims 2 to 8 and 12 and/or the waxes are as defined in claim 9 and/or claim 10.
- 20. A method according to claim 18 or 19 in which the montan wax is used in an amount in the range 10 to 20% by weight of the emulsion, the hydrocarbon wax is used in an amount in the range 20 to 40% by weight of the emulsion and the emulsifier system is used in an amount in the range 0.5 to 6% by weight, preferably 1 to 2.5% by weight of the emulsion.

20

25



1/2

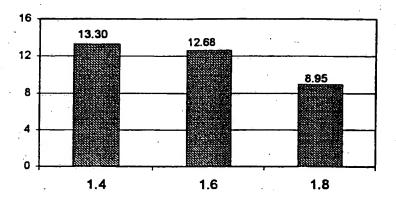


Figure 1

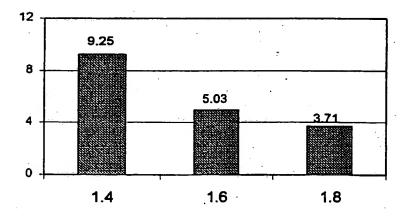


Figure 2

534 Rec'd PCT/PTC 07 JUL 2000

2/2

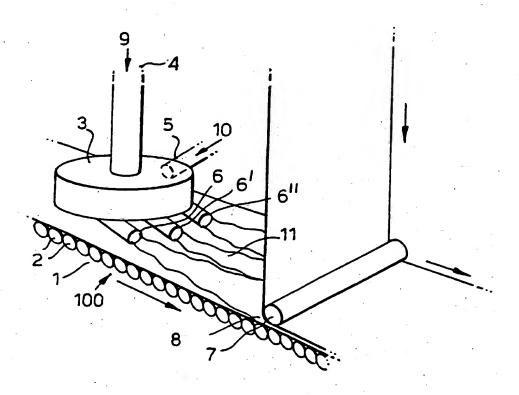


Figure 3

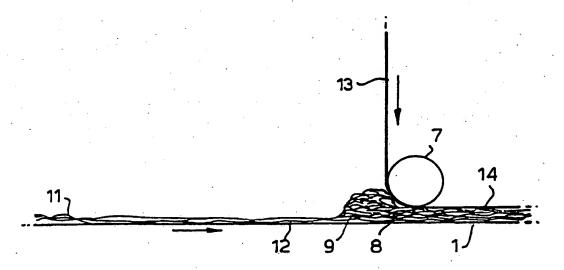


Figure 4

534 Rec'd PCT/PTC 07 JUL 2000

INTERNATIONAL SEARCH REPORT



PCT/GB 99/00064

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C04B28/14 C08L91/06

C04B111:27

//(CO4B28/14,24:08,24:22,24:32,24:36),

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols) IPC 6 C04B C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 695 553 A (T. CLARET, ET AL.) 9 December 1997 see claim 1	1,4,9, 11,13, 14,16-20
A,P	WO 98 09925 A (MOBIL OIL) 12 March 1998 cited in the application see page 4, line 1-11; claims 1,8	1,3-6
Α	US 4 315 957 A (HOECHST AG) 16 February 1982 see column 3, line 6-10 see column 3, line 35-45; claim 1	16,18,20
	-/	
		n e

X Further documents are listed in the continuation of box C.	Pätent fämily members are listed in annex.			
"A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filling date	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention			
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the			
"O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed	document is combined with one or more other such docu- ments, such combination being obvious to a person skilled in the art. "8" document member of the same patent family			
Date of the actual completion of the international search	Date of mailing of the international search report			
22 April 1999	03/05/1999			
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk	Authorized officer			
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Daeleman, P			

1



INTERNATIONAL SEARCH REPORT

Intel onal Application No PCT/GB 99/00064

C (C:-	PCT/GB 99	, 0000 4
	Ation) DOCUMENTS CONSIDERED TO BE RELEVANT	12
Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A,P	PATENT ABSTRACTS OF JAPAN vol. 098, no. 008, 30 June 1998 & JP 10 067980 A (MITSUBISHI CHEM CORP), 10 March 1998	16
*** * ********************************	see abstract US 5 437 722 A (L. BORENSTEIN)	
	1 August 1995	
	cited in the application	
\	DATABASE WPI Week 8635 Derwent Publications Ltd., London, GB;	
	AN 86-229176 XP002100852	
	& JP 61 159494 A (SANWA KAGAKU KK) see abstract	* **
		· · · · · · · · · · · · · · · · · · ·
*		*
		,
		*
. ,		
		á.
·		
	*	

INTERNATIONAL SEARCH REPORT

Information on patent family members

Inter anal Application No PCT/GB 99/00064

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
US 5695553	Α	09-12-1997	CA	2184041 A	24-02-1998
WO 9809925	Α	12-03-1998	AU	4125397 A	26-03-1998
US 4315957	A	16-02-1982	DE AU AU BR EP JP JP ZA	2926197 A 535960 B 5972480 A 8004068 A 0022212 A 56010559 A 63058193 B 8003853 A	15-01-1981 12-04-1984 08-01-1981 21-01-1981 14-01-1981 03-02-1981 15-11-1988 29-07-1981
US 5437722	A	01-08-1995	AU CA EP JP AT AU DE	676965 B 2116483 A,C 0669377 A 8337459 A 175707 T 1637595 A 69507174 D	27-03-1997 26-08-1995 30-08-1995 24-12-1996 15-01-1997 24-10-1996 25-02-1999

